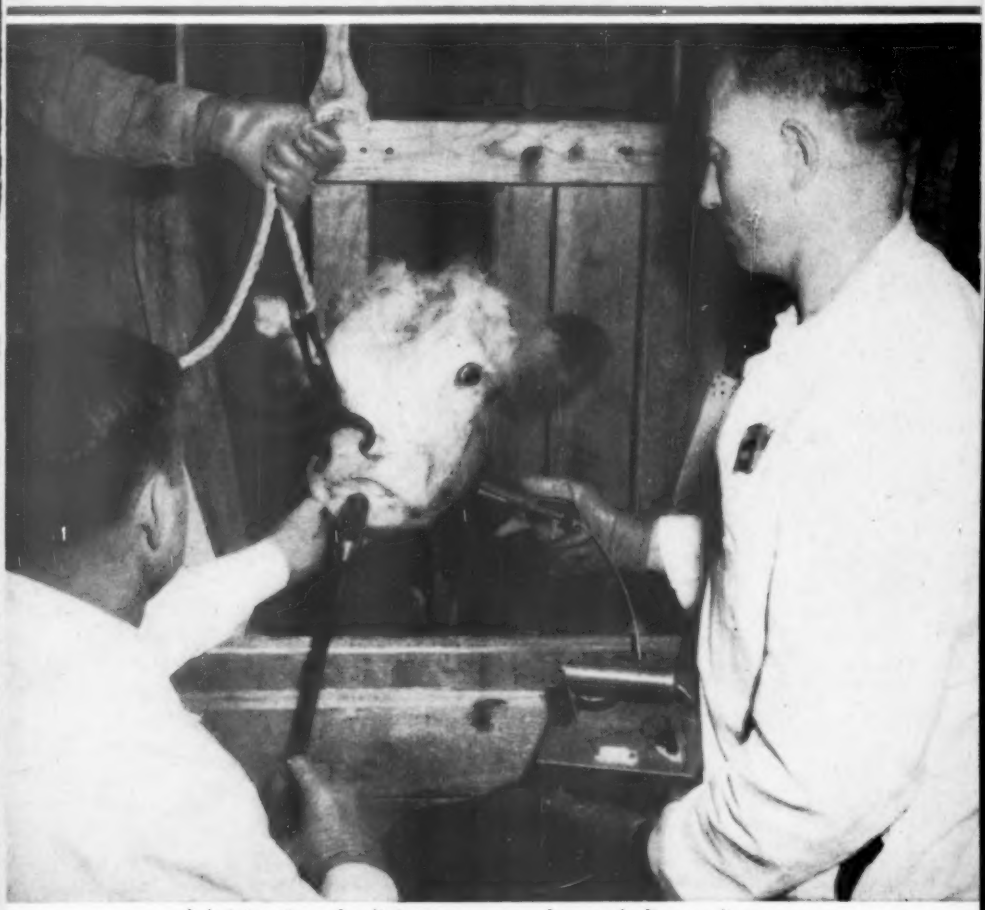
NUCLEONICS

TECHNIQUES AND APPLICATIONS OF NUCLEAR SCIENCE AND ENGINEERING

August, 1951



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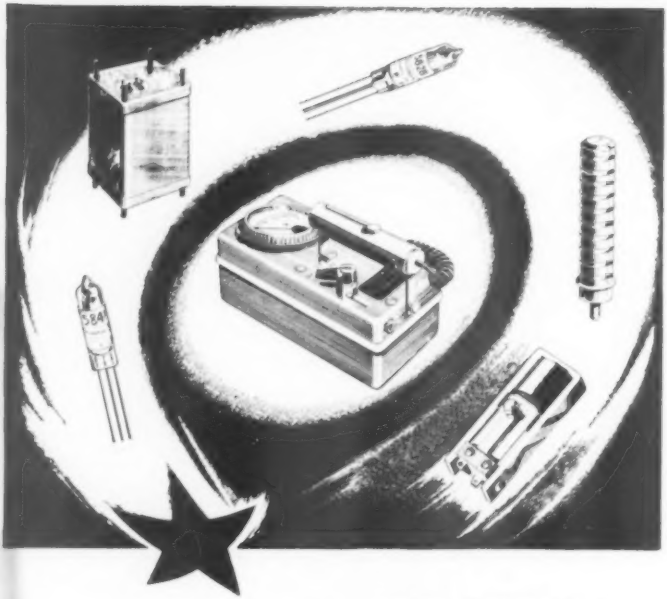

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NUCLEONICS

AUGUST · 1951

Vol. 9, No. 2

THE COVER shows how radioisotopes are administered orally to a steer immobilized in a metabolism stall (see article on page 38). A balling gun is used to discharge capsule containing isotope on rear of steer's tongue. Animal's mouth is then held closed until capsule is swallowed. Its movement into the stomach is followed by monitor at right

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International Exchange of Information

The times we are living in are such that many responsible officials feel it is safer to overclassify than underclassify information. They can't be criticized by Congress or the military for doing this.

This feeling is strongly in evidence, particularly in the case of exchanging information with foreign nations. After all, it is felt, what can we gain by "giving information away?"

In the atomic energy field, in addition to restrictions on classified information, exchange of unclassified information is not as free as it might be—even with the United Kingdom and Canada. In fact in the case of England, it is reported that representatives of that country were rebuffed recently on a request for information on one of our large particle accelerators, a completely unclassified machine.

"We are exchanging information on such a limited front," says former AEC Commissioner R. F. Bacher, "that it is almost certain that the same problems are being studied in the U. S., Britain and Canada. If the aim is mutual progress, such a policy makes little sense."

One member of AEC's General Advisory Committee goes further and says, "If we had more extensive international exchange of information, the U. S. would benefit the most by it since we have the manpower and personnel to make use of this information whereas the others don't."

One area in which more freedom is needed is in connection with unclassified scientific meetings in this country. Recent experience has shown that an invitation can no longer be extended to a foreigner solely because of his scientific excellence. Now his associations must also be beyond question.

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This attitude is not only causing scientists in this country considerable embarrassment, but it is depriving them of the intellectual stimulation which they would receive by contact with their colleagues from other parts of the world. If we are seeking progress in science, it is obvious that we are taking the wrong tack.

Foreign Sale of Isotopes

One important step in the direction of furthering international cooperation in science was announced by the AEC last month. For the first time since foreign distribution of isotopes was initiated in 1947, there will be no limit on the type of isotopes which can be sold abroad (with the exception of tritium).

Previously, foreign countries were limited to the purchase of 26 isotopes, useful primarily in the life sciences. Now the total number available will be 99, including the heretofore banned industrially useful isotopes (see p. 80).

This change in policy was no doubt accelerated by the fact that nuclear reactors are now in operation in at least four countries outside the U. S. To date, we have made a total of about 1,135 isotope shipments abroad, whereas the United Kingdom made 1,291 foreign shipments in 1950 alone.

Electric Power Industry and the AEC

It was once stated that the efficiency of a committee varies inversely as the number of members. This can be expanded to say that the efficiency of an organization varies inversely with the number of committees which advise it. However, frequently an agency finds it necessary to use advisory committees

(Continued on page 71)

Random Notes

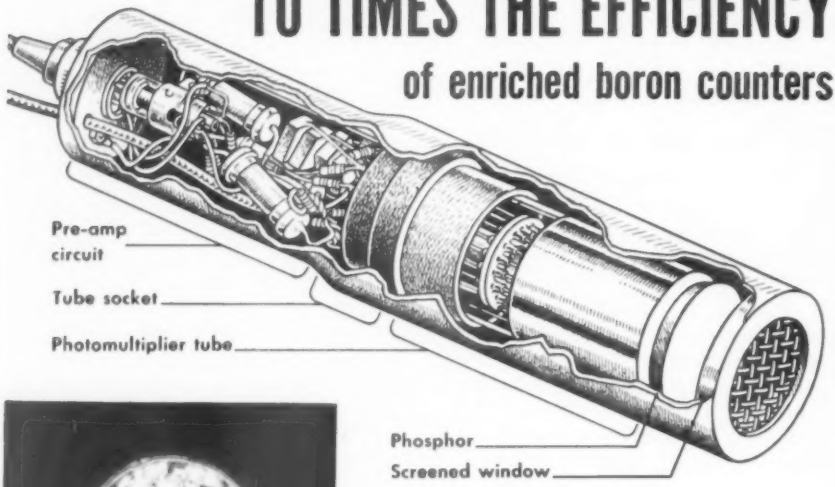
• *Construction is expected to start this summer on the new heavy-water reactor at Chalk River, Canada. It will be more powerful and have a higher neutron flux than the NRX pile at Chalk River, and will be adapted to extend greatly the range of possible research, as well as to increase production of plutonium and radioisotopes. During the past year, the NRX reactor operated at a power level somewhat above the designed value of ten megawatts, with a maximum flux density of approximately* 6×10^{13} *neutrons per* cm^2 *per sec.*

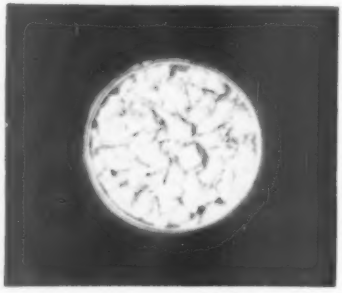
The plant at Chalk River designed for the separation of uranium-233 from irradiated thorium has been in operation for a number of months now and has produced preliminary results which are considered to be satisfactory.

• Research with kilocurie radiation sources and with waste fission products (see p. 10) may turn up new uses of radiation. In addition, these sources will be applied to problems which are presently handled by other means, e.g. X-ray machines. If the sources do prove to be technically satisfactory in

(Continued on page 71)

NEW G-E PROBE detects thermal neutrons at 10 TIMES THE EFFICIENCY of enriched boron counters





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GENERAL ELECTRIC

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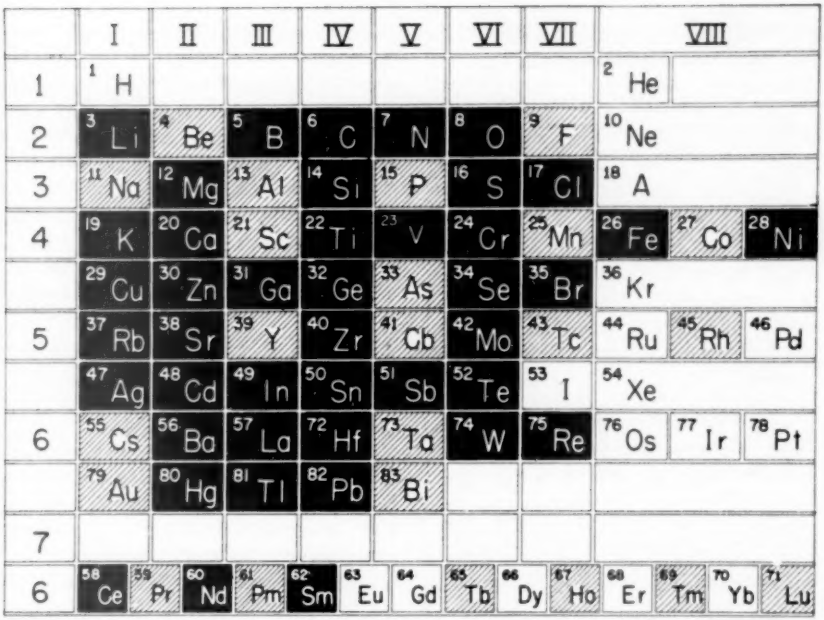
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1 H He
2 Li Be B C N O F Ne
3 Na Mg Al Si P S Cl A
4 K Ca Sc Ti V Cr Mn Fe Co Ni
Cu Zn Ga Ge As Se Br Kr
5 Rb Sr Y Zr Cb Mo Tc Ru Rh Pd
Ag Cd In Sn Sb Te I Xe
6 Cs Ba La Hf Ta W Re Os Ir Pt
Au Hg Tl Pb Bi
7
6 Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu
X ELEMENTS THUS FAR PROCESSED
X ELEMENTS NOT YET PROCESSED
X ELEMENTS WITH NO DETECTABLE ISOTOPES

FIG. 1. Portion of periodic chart of the elements shows extent of stable isotope program: 43 elements having 177 isotopes have been processed

ENRICHED STABLE ISOTOPES: Five Years of Production and Use*

By C. P. KEIM

Director, Stable Isotope Research and Production Division
Oak Ridge National Laboratory, Oak Ridge, Tennessee

PRIOR TO 1946 the electromagnetic isotope separation facilities at Oak Ridge were employed to separate U^{235} from the more abundant U^{238}. During this same time, the gaseous diffusion plant for enriching U^{235} was placed in operation and superseded the electromagnetic process. But a small part of the electromagnetic separation facilities have been kept in operation for research and development of the process and for production of enriched stable isotopes.

The success of this stable-isotope separation program is portrayed by Fig. 1. Of 61 elements having about 255 known natural stable isotopes (those

* This article is based on work performed for the AEC by Carbide and Carbon Chemicals Co., a division of Union Carbide and Carbon Corporation.

ENRICHED STABLE ISOTOPES
have been used in research. . . .

To measure total, absorption, and scattering cross sections for thermal neutrons. Almost 100 isotopes have been studied in this way.

To measure cross sections for proton and deuteron reactions; examples are Mo, Zn, Cu, Ni.

To identify the masses of radioisotopes produced from enriched stable isotopes.

To investigate the radioactivities, decay schemes, and half-lives of artificially produced radioisotopes.

As starting materials for pile and cyclotron produced radioisotopes.

In neutron diffraction experiments.

In studies on the rare-earth nuclides.

To study beta-ray spectra (K^{40}, and artificial Be^{10} separated from Be^{9} were used in this way).

In photographic plates (Li^{6}) for measuring the energy of fast neurons.

To establish more firmly weak natural radioactivities of isotopes such as Rb^{87}, In^{115}, Sn^{124}, Sm^{147}, Re^{187}.

To determine threshold voltages for photoneutron reactions in Zn, Li, Mg, and Si.

To determine isotope shift, hyperfine structure, nuclear spin values and magnetic moments.

To determine nuclear energy levels for a number of isotopes of different elements—K, Pb, Bi, Tl, Se, Si, Mg, Ag, Al, and others.

To study superconductivity of Hg and Sn, bringing theory and experiment closer together.

elements above Bi in the chart having only natural radioisotopes are not being considered), 43 elements, or 70%, having 177 isotopes have been processed. From January, 1946 to April, 1951, 879 collections of enriched isotopes in milligram and gram amounts have been completely prepared. From this stock, 1,146 shipments have been sent to 58 different laboratories for research purposes. Of the shipments made, 703, or 61% of the total, went to AEC laboratories, while 443, or 39%, went to university and industrial laboratories. It is interesting to note, however, that 80% of the users were non-AEC laboratories.

At least 200 reports have appeared in the unclassified technical literature reviewing research accomplishments with these enriched isotopes. Many classified reports have also been written. The scope of this research is indicated by the partial listing of the research applications of enriched stable isotopes that appears on this page.

Production Facilities

Stable-isotope enrichment is carried out in mass spectrographs called calutrons at Oak Ridge. Except for size, there is little difference between a laboratory mass spectrometer or mass spectrograph and its production companion, the calutron. Such machines all produce ions of the desired isotopes at the source, accelerate these ions by means of electric fields, separate the paths of travel of the different isotopic ions in magnetic fields, and measure or retain the isotopes at collectors at the end of their travel, all components operating in a vacuum. The primary difference between the laboratory mass spectrometer and the production instrument is that in production it is intended to collect usable quantities of desired isotopes, while the mass spectrometer is used primarily for detection and measurement, being able to produce

only minute quantities of the isotopes.

The basic equations for the calutron are those so characteristic of the Dempster-type mass spectrometer. Looking at the joint effects of electric and magnetic fields

$$m/e = H^2R^2/2V$$

where m = mass of the ion, e = charge carried by the ion (the singly charged ion is usually most prominent), H = strength of magnetic field, R = radius of ion path from source to collector, and V = accelerating voltage at the source. It is apparent that any ion of mass m, carrying charge e, can be made to travel the path of radius R if the magnetic field H and accelerating voltage V can be chosen to satisfy this equation.

It has been found by experience in electromagnetic isotope separation that usually it is only necessary to alter the magnetic field when changing operations from an element of atomic mass m_1 to an element of mass m_2. Thus, the ratio of the masses equals the ratio of the squares of the magnetic fields.

The effectiveness of the resolution of isotopes in electromagnetic separation is shown in Fig. 2, where an oscillogram

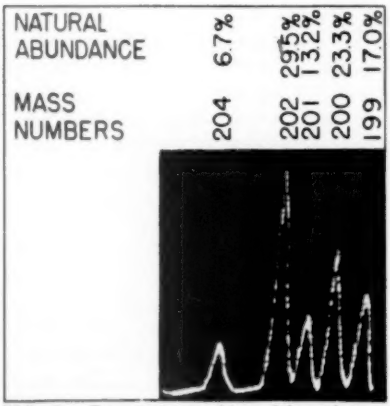

FIG. 2. Resolution achieved in calutron separation is shown by this oscillogram of five of the isotopes of mercury. Note sharpness of peaks; height corresponds to natural abundance

pictures the isotope peaks and valleys of five of the mercury isotopes. This high resolution was possible because the Hg vapor pressure within the vacuum chamber of the calutron was greatly reduced by cooling. The two remaining isotopes, Hg^{196} and Hg^{198} are not shown because the oscilloscope screen could not accomodate all seven isotopes at the same time for photographing. The relative amounts of each isotope in natural mercury is indicated by the height of the peaks. It can be seen why it is possible to enrich Hg^{202} to at least 98.5%.

Production Techniques

The problem of separating isotopes electromagnetically may be divided into several phases.

1. For each element a suitable charge material (giving adequate vapor pressure at a reasonable temperature) for the calutron ion source must be selected and obtained or prepared.

2. The best operating conditions (vacuum, magnetic field, and accelerating voltage) for the calutron must be determined for the charge material being used.

3. The separated isotopes must be quantitatively recovered from the collectors, then chemically purified.

4. A spectrochemical analysis must be made to determine if the isotopic material is sufficiently pure chemically.

5. A suitable sample for a mass analysis in the mass spectrometer must be prepared and the isotopic distribution in the collected sample determined.

Of the isotopes not yet processed, the enrichment of those of ruthenium, palladium, osmium, iridium and platinum depend on the successful preparation of suitable charge materials. Most of the rare-earth isotopes await the chemical separation of sufficient quantities of the rare-earth elements themselves, from which to make charge

materials. Encouraging progress is being made in our chemistry laboratories on these problems, and the rare-earth elements cerium, neodymium, and samarium have been processed. Gadolinium isotopes will be collected when calutron time becomes available. The noble gases, neon, argon, krypton, and xenon also await calutron time.

After a collection in the calutron is completed, the source, collector and other units must be removed from the vacuum chamber between the poles of the electromagnet, taking precautions required by the toxicity and, in some cases, the radioactivity of the material being processed. The isotope containers are handled in a way to avoid loss and contamination, and are transferred to the chemistry laboratory, where the isotopes are quantitatively removed by distillation or dissolving. Obviously, from here on, chemical purification procedures differ with each element.

From 1945–1951, over 800 samples of isotopes of 44 elements were chemically refined from contaminants as a result of newly developed or newly adapted methods of quantitative separation. Notable improvements in these chemistry techniques were with zirconium, hafnium, gallium, rhenium, beryllium, indium, and the rare earths—cerium, neodymium, samarium and gadolinium.

Another important problem is in the mass analysis of the enriched isotope product. The mass spectrometer laboratory, under R. F. Hibbs and J. W. Redmond, has kept analysis techniques abreast of the isotope separation program. The analytical mass spectrometer often requires a different charge material and different techniques than the calutron for the same element. Continued research is necessary until all enriched isotopes can be reliably analyzed for their isotopic content.

In five years, the AEC Stable Isotopes Division has . . . .

- Processed 879 samples of 117 isotopes
- Made 703 shipments to 11 AEC laboratories
- Made 443 shipments to 47 non-AEC laboratories

Typical Results

Some typical isotopes which have been enriched electromagnetically are listed in the table on the next page. The isotopes of carbon are not usually enriched in the calutron except to obtain collections for particular use, such as 99.99% C^{13}. Very exact chemistry is necessary to avoid contaminating such pure C^{13}. K^{40} is a beta emitter, and it has many interesting research applications as it is enriched successfully to higher and higher concentrations. By using pile-irradiated potassium, in which the K^{40} natural content of 0.01% is augmented by some artificially produced K^{40} from the K^{39} (n,γ) K^{40} reaction, we hope to obtain some enriched K^{40} well beyond the 7% material thus far produced from the normal potassium starting material.

For some reason the calutron enriches calcium isotopes very effectively, as may be seen from the enriched abundances obtained. Iron has been processed principally to obtain enriched Fe^{58}, which is used to produce the 40-day Fe^{59} in the pile by an (n,γ) reaction, with only a small amount of the 4-year Fe^{55} being produced from the reduced abundance of Fe^{54}. Up to now we have accumulated several hundred grams of 99% Fe^{58}, which we wish someone could use to good advantage.

Gallium is an element used in current bone-cancer research. To separate the isotopes, a great deal of basic chemistry was done in cooperation with the Oak

Some Typical Isotopes Processed and Isotopic Enrichments Attained

Element	*Isotope mass*	*Natural abundance (%)*	*Enriched abundance (%)*
Lithium	6	7.39	99.4
	7	92.61	99.91
Carbon	12	98.9	99.99
	13	1.1	7.52
Potassium	39	93.3	99.94
	40	0.011	7.75
	41	6.7	98.94
Calcium	40	96.96	99.97
	42	0.64	61.4
	43	0.15	59.9
	44	2.06	96.2
	46	0.0033	7.3
	48	0.19	83.9
Iron	54	5.81	93.27
	56	91.64	99.70
	57	2.21	87.29
	58	0.34	79.8
Gallium	69	60.2	98.42
	71	39.8	98.08
Zirconium	90	51.46	98.00
	91	11.23	86.89
	92	17.11	95.38
	94	17.40	92.8
	96	2.80	89.48
Cerium	136	0.193	16.6
	138	0.250	8.9
	140	88.48	99.25
	142	11.07	87.4
Neodymium	142	27.13	93.00
	143	12.20	83.93
	144	23.87	93.45
	145	8.30	78.60
	146	17.18	95.60
	148	5.72	89.85
	150	5.60	94.76
Samarium	144	3.16	72.13
	147	15.07	81.63
	148	11.27	76.01
	149	13.84	71.53
	150	7.47	74.09
	152	26.62	89.90
	154	22.53	92.10
Hafnium	174	0.18	7.85
	176	5.30	48.46
	177	18.47	61.71
	178	27.10	80.91
	179	13.84	46.57
	180	35.11	93.96

Ridge cancer research hospital. Since the 14-hr Ga^{72} is the radioisotope of particular value, the possibility is being evaluated of using 98–99% enriched Ga^{71} in the pile (n,γ) reaction to produce Ga^{72}.

Zirconium isotopes were produced from high-grade zirconium which had been chemically freed from hafnium. Using hafnium-free zirconium was desirable because it was found that doubly-charged Hf^{180} entered the same collector in the calutron as the singly-charged Zr^{90}, thereby severely contaminating it.

Very pure hafnium was also obtained from the zirconium-hafnium chemical separation process. From this, a charge compound for hafnium isotope enrichment was obtained. A by-product of interest and importance was a sizeable amount of 95%-pure hafnium metal.

The enrichment of Sm^{147} from 15 to 81% made it possible to establish the alpha activity of that isotope. Although the isotopes of indium are not included in the table, the enrichment of In^{115} made it possible to measure its radioactivity also.

Availability of Stable Isotopes

Catalogs and further information about the procurement of enriched stable isotopes can be obtained from the Isotopes Division, U. S. Atomic Energy Commission, Oak Ridge, Tenn. —END

Thousand-curie source of radiotantalum glowing inside a steel pipe before transfer to protective lead shield. This is the bottom of canal into which materials irradiated in the Brookhaven reactor are deposited

Use of Kilocurie Radiation Sources

Applications of radioactive waste materials from reactors are now being studied. Brookhaven is producing sources of 1,000–2,000 curies to simulate radiations from these wastes. Sources will be used for research and for industrial irradiations

By BERNARD MANOWITZ

Reactor Science and Engineering Department
Brookhaven National Laboratory, Upton, N. Y.

DURING THE PAST YEAR, a program has been under way to study the possibilities for the utilization of the radioactive wastes produced as by-products of plutonium production.* The decision on whether the Atomic Energy Commission will go to the expense of making these wastes available in usable form will be based on the possible applications of these materials and on the resulting interest.

A program has been set up under Brookhaven National Laboratory, Upton, L. I., N. Y., to get further information on applications. At the outset, kilocurie radiation sources are being produced in the Brookhaven reactor for use in simulation of the radiation which would be produced from the waste products. These special sources will be used in three ways:

1. At Brookhaven in the laboratory's own investigations into applications of the radiation.

2. At Brookhaven as irradiation services performed for non-AEC organizations interested in determining radiation effects on various materials.

3. At the laboratories of various AEC contractors where research on

* Industrial Utilization of Fission Products, NUCLEONICS **8**, No. 5, 5 (1951).

specific problems will be carried out.

Work on the direct utilization of the reactor waste products will be more involved than the above programs because special separation methods and facilities will have to be developed; it is expected that such work will be carried out at Brookhaven in the near future.

Radiation Sources

Although the gross fission products emit both β- and γ-radiation, we have produced sources which are primarily γ-emitters. These are cobalt-60 with a half-life of 5.3 years, γ's of 1.33 and 1.17 Mev, and a thermal-neutron absorption cross section of 34 barns; and tantalum-182 with a half-life of 117 days, γ's of 1.22, 1.13, 0.22, and 0.15 Mev, and a thermal-neutron absorption cross section of 21 barns.

Co^{60} has the advantage of a long half-life. In comparison, Ta^{182} has a short half-life and will have to be returned to the pile every six months to be irradiated again. Despite this, however, it has the advantage of giving a higher specific γ-flux. Also, its lower cross section will not cause it to "poison" the pile as much as the Co^{60}, benefiting the pile neutron economy.

The cobalt was purchased from the Belmont Smelting and Refining Works (New York) and was fabricated into tubular shape by the Technical Fabrications Company (Cambridge, Mass.). Two of these tubes (each about 6.6 in. in length, 2.2. in. O.D. and 1.7 in. I.D.) were placed end-on-end within two concentric aluminum tubes 13.5 in. long, 2.3 in. O.D. for the outer tube, and 1.5 in. I.D. for the inner tube. The ends of the tubes were welded so the Co and Al would be one unit (Fig. 1).

The tantalum was purchased from the Fansteel Metallurgical Co. (Chicago, Ill.) in 0.060-in. sheets and was rolled into tubular shape and inserted between aluminum tubes, the ends of which were also welded.

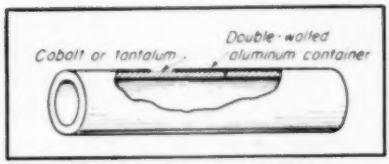

FIG. 1. Source container

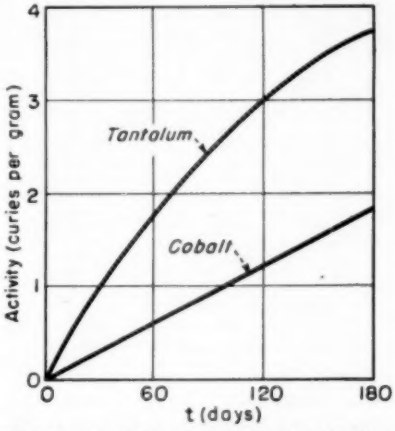

FIG. 2. Activity produced on irradiation in pile of 1 gm of material in flux of 3×10^{12} $n/cm^2/sec$

The complete units (cobalt plus aluminum, and tantalum plus aluminum) were placed in the pile and irradiated for 60 days. Irradiation time was determined by the equation

$$S = \frac{0.6\sigma\phi}{3.7 \times 10^{10} A}(1 - e^{-0.693t/T})$$

where S = curies/gm, ϕ = thermal neutrons/cm^2/sec, σ = activation cross section in barns, A = atomic weight of irradiated material in grams, T = half-life of activity produced in days, and t = irradiation time in days. Using this equation for 1 gm of irradiated material and a flux of $3 \times 10^{12} n/cm^2/sec$, we drew the curves shown in Fig. 2.

An average amount of 2,650 gm of cobalt and 1,400 gm of tantalum was irradiated per tube so that one calculates from the curves that 1,643 curies

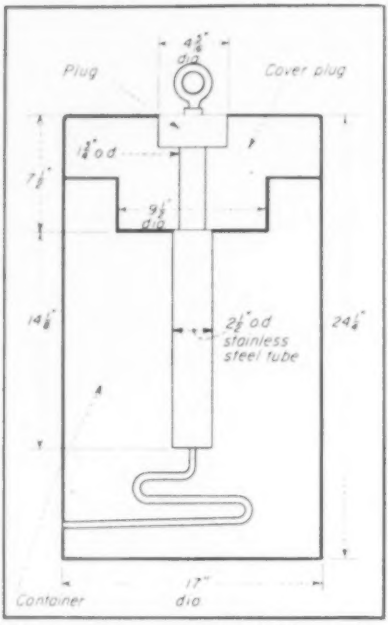

FIG. 3. Pig assembly for holding radiation source and irradiating samples

of cobalt and 2,450 curies of tantalum were produced by a 60-day irradiation. However, when one puts a large poison in the pile, a flux depression of magnitude difficult to estimate is created in the immediate area. In addition, if the tubes are placed close to other experiments of large cross section, or close to control rods, a shadow effect is produced. Some correction has been made for flux depression but, in view of the unknown involved, we prefer to designate the cobalt sources nominally as 1,000 curies and the tantalum sources as 2,000 curies.

Five sources—three Co^{60} and two Ta^{182}—have been produced. Four more—two of each—are in the pile.

Source Container

Because of the intense gamma radiation emitted by the sources, a lead pig had to be designed to serve as a shield. Figure 3 shows such a shield; it is also used to hold the sample for irradiation.

A sample tube approximately 12 in. long and 1¼ in. I.D. is attached to the plug as shown in Fig. 4. This tube places the limitation on the maximum sample size which can be irradiated.

In practice at Brookhaven, the sources, discharged from the reactor into a canal, are picked up with long-handled tongs and placed in the container. The cover is lowered into place with a chain fall, and the entire assembly is removed by a hoist. After the canal water has drained from the pig, the drain plug is inserted.

To irradiate a sample, the small plug in the top of the cover is removed with a long pole (an automatic device for removing the plugs is under design). The sample is placed in the sample tube, and the plug and tube are set back into the pig assembly.

The pig is made of lead with a total weight of 2,300 lb. With a source of 1,600 curies set in position in the pig, the radiation dosage at the outside surface has been found to be 25 mr/hr.

Radiation Field

The tubular form of the sources makes available an intense and uniform radiation field. The intensity of a β-γ field is ordinarily described in terms of the roentgen-equivalent-physical (rep) whereas amounts of radioactive materials are described in terms of curies. The actual relationship between curies of fission products and rep of β-γ radiation will be a function of the energy of the β-γ particles and of the geometry used.

However, for purposes of crude approximation, we can make use of the empirical information obtained with the Brookhaven tantalum source. It was found that a 2,000 curie source produced a radiation field within the tube of about 5×10^5 rep/hr. Work is now in progress on the calibration of

FIG. 4. Inserting sample tube in holder for fastening to steel plug (left)

the sources by means of ionization chambers, photographic film, and chemical dosimeters.

The work of Dunn* and others has indicated that a dose of 5×10^5 to 10^6 rep is sufficient to kill many bacteria, inactivate many enzymes, and, in general, is a reasonable dosage to consider for being ultimately useful in industries concerned with biological reactions.

Work at Brookhaven and elsewhere has indicated that doses of $5\text{–}50 \times 10^6$ rep are required to effect many chemical reactions.

Working within the pattern of available knowledge, we feel it is not inconceivable that a million-curie source would be sufficient to service a company sterilizing or preserving a ton of material per day whereas 10–100 million curies would be required to process a ton of chemical products per day.

It would be possible to obtain these amounts of radioactivity from the waste streams of presently operating or presently conceived reactors.

At Brookhaven, it is possible that sources up to 10,000 curies will be produced in the reactor. The only limitation will be the available flux of the reactor.

Source Availability

Organizations interested in having samples irradiated by the Brookhaven source can obtain further information by writing the Director's office at the laboratory. Samples will be accepted in liquid, gaseous, or solid form with the size limitation mentioned previously.

It is expected that the cost will be based on the total irradiation time requested. For an eight-hour day, the cost will be approximately $40, plus a $25 servicing charge.

With regard to work being done with these sources elsewhere, a Co^{60} source has already been sent to the University of Michigan. It is expected that sources will go also to MIT, Yale, Columbia, and the General Electric Company at Schenectady. All of these organizations have contracts with the AEC for this work.

Columbia is studying methods of food preservation and possible food toxicity from irradiation. MIT is investigating the radiation dosage necessary to kill bacteria and other organisms which contaminate food products. Yale is working on determining the effects of gamma radiation on chemical reactions of gases.

It is to be expected that this entire program will, over the next several years, throw light on possible applications of reactor waste products. If such uses are found to be extensive and the price of these products can be made low enough, our industrial technology will benefit and part of the AEC's waste problem will be solved. —END

* C. G. Dunn, *et al.*, *J. Applied Phys.* **19**, 605 (1948)

Determination of Radium or Radon in Gases, Liquids or Solids

As little as 10^{-14} gm of radium or 10^{-20} gm of radon may be measured by this method of counting the radon activity. Chemical techniques for sample preparation, construction of the ionization chamber, and circuitry are described in detail

By J. E. Hudgens, R. O. Benzing, J. P. Cali, R. C. Meyer and L. C. Nelson

Radiochemical Branch, New Brunswick Laboratory, U. S. Atomic Energy Commission New Brunswick, New Jersey

QUANTITATIVE DETERMINATION of radium in concentrations of 10^{-14} gm, or more, of radium per gram of sample may be accomplished in several ways. These may be classified according to the method used to determine the activity of radon and its decay products.

An integration type of detector was used for radon determination by Rutherford (*1*) who used a sensitive electrometer connected to a brass cylinder to measure the ion current produced by radon and its products. The inherent criticalness of the components of this arrangement, as well as the large fluctuations in the background which are observed, make it undesirable for determining small amounts of radium or radon. The error contributed by background fluctuations has been minimized by connecting the electrodes of two identical, oppositely charged chambers to an electrometer (*2-6*).

The disadvantages of the electrometer type of detection have been discussed in some detail by Curtiss and Davis (*7*) who described a pulse type of detection unit. Fineman *et al.* (*8*) refined the apparatus and technique used by Curtiss and Davis and studied the effect of a number of variables on the determination. These authors consider the lower limit of their apparatus to be about 10^{-12} gm of radium.

The principal difference between the apparatus and technique described here and those of Davis and Curtiss and Fineman *et al.* is in the chamber design and the amplification and recording system.

Stability of the high-voltage gain and of the scaler triggering has been achieved by filtering the voltage supply and by attention to good construction practice. The system described is relatively stable and requires little maintenance or attention by the operator. The limit of detection is about 10^{-14} gm of radium, or 10^{-20} gm of radon.

The electronic circuitry has been so designed that it may be used for purposes other than radium determination.

Sample Preparation and Chamber Filling

In analyzing samples of relatively low radium content the weight of radon which must be transferred to the chamber is usually about 10^{-18} to 10^{-20} gm. Analysis of ore samples may involve transfer of radon weights greater than this by several factors of ten.

The gas used as a source of ion pairs in the alpha counting chamber must be relatively free of impurities, espe-

cially of water, acid vapors, oxygen and carbon dioxide. The importance of carbon dioxide can be decreased by using one of the inert gases (helium or argon). A partial pressure of 0.1 atmosphere of carbon dioxide to 0.9 atmosphere of argon results in large gas amplification factors in chambers fitted with suitable collecting electrodes (*9*). Several methods of removing impurities may be used, and while the combination of absorbents given below is satisfactory, other methods may be used with equal success.

The apparatus used to fill the chambers is shown in Fig. 1. The flask containing the radon is connected to the system at the ball and socket joint Q. The flask shown in the figure is nearly identical to that described by Evans (*2–4*) and is used for liquid samples. Solid samples may be put into solution and placed in this flask or may be treated in a manner similar to that described by Evans. Commercial nitrogen gas of the water-pumped or oil-pumped variety may be used to sweep radon from the flask into the counting chamber.

The nitrogen (with the radon) is swept through the solution up through the condenser, where the water is condensed and partially removed from the gas stream; through a tube containing copper turnings which are heated to approximately 500° C; over a drying tube containing magnesium perchlorate; over a tube containing ascarite; and finally into the counting chamber which is connected at R.

If samples containing large concentrations of acid or carbon dioxide are to be run, a second magnesium perchlorate trap should be installed between the ascarite trap and the counting chamber.

Argon gas may be used with equal facility as a counting atmosphere. However, since the cost of argon is higher, and since no appreciable difference in the counting characteristics were observed when the two gases were compared, nitrogen is used exclusively in this laboratory. The ascarite serves the dual purpose of removing carbon dioxide from the gas stream as well as removing any acid vapors which may be carried over from the acidic solution of solid samples.

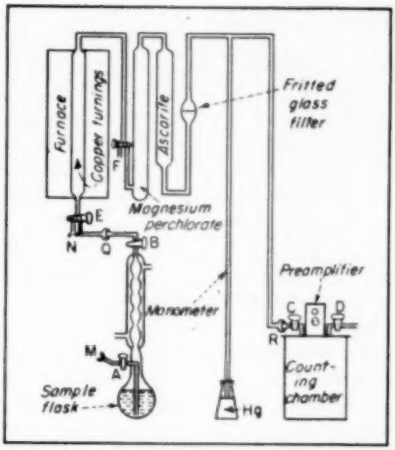

FIG. 1. Apparatus for purifying gas and filling ionization chamber

Preparation of radium solution. A well mixed sample is weighed into a porcelain dish and ignited overnight at 550° C in a muffle furnace. The dish and sample are then reweighed to determine the moisture and organic matter lost. The sample is then pulverized and a known weight (usually 1.5 gm) mixed with 30 ml of 8*M* nitric acid. After boiling the mixture for about 15 min, 50 ml of water are added and the solution filtered through No. 42 Whatman filter paper. The filter paper and any insoluble residue is ignited in a platinum crucible and then treated with about 20 ml of hydrofluoric acid. The hydrofluoric acid is evaporated and the residue fused for 15 min with about 15 gm of sodium carbonate. The fused mass is dissolved in 50 ml 8*M* nitric acid, filtered, and the filtrate added to

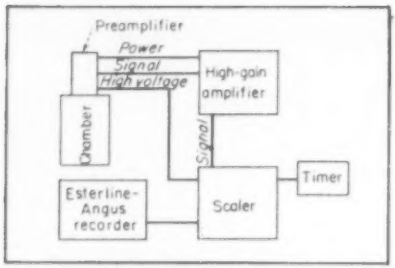

FIG. 2. Block diagram of detection and recording apparatus

that obtained from the first nitric acid filtration.

If solution of the sample by the carbonate fusion is not complete, a second hydrofluoric acid treatment and sodium carbonate fusion is performed. The combined filtrates are diluted to a known volume in a volumetric flask and a suitable sample aliquot transferred to the distillation flask shown in Fig. 1. Nitrogen is then bubbled through the hot solution in the flask to remove any radon which may have accumulated since the solution of the sample. Stopcocks A and B are closed and the flask set aside to allow the radon to grow into partial or total equilibrium with the radium.

Transfer of radon to chamber. The flask, after a sufficient time has been allowed for radon growth, is attached to the vacuum system; the counting chamber which is to be used in the determination is attached at R. The system up to stopcock B is then evacuated to a pressure of about 50 μ with a mechanical vacuum pump. Stopcock D to the pump is then closed and the stopcock leading from the flask opened to allow the radon to be swept into the chamber. A slow stream of nitrogen is then allowed to flow through stopcock A, through the solution, over the absorption tubes, and into the chamber.

The gas flow is regulated so that about 25 min is required to raise the pressure in the system to atmospheric. During the filling process the solution in the flask is heated with a heating mantle to boiling and maintained at this temperature until the filling process is completed. When atmospheric pressure has been reached in the system, stopcocks A, B, and C are closed and the

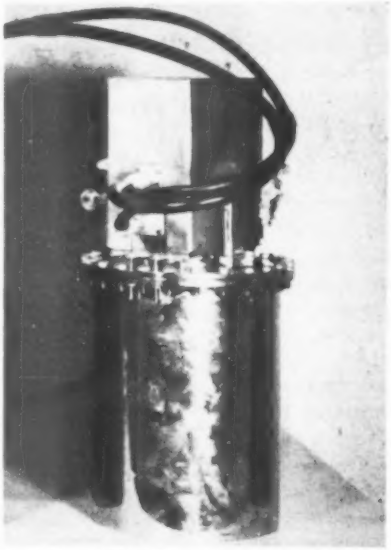

FIG. 3. Ionization chamber with preamplifier on top.

chamber removed from the apparatus.

After determination of the radioactivity in the chamber, the chamber is again attached to the purification system and nitrogen flushed through the system and the chamber by attaching a nitrogen supply at the ball and socket joint N. Stopcocks E and F facilitate the removal of oxygen from the tube containing the copper turnings. The next determination may be made in 30 minutes which is usually sufficient time for all the radon to be removed.

A relatively small number of gas purification systems are required to permit a large number of analyses to be performed, since the same purification

system may be used for a relatively large number of ionization chambers and sample flasks.

The Apparatus

A block diagram of the arrangement of the different components is shown in Fig. 2. Figure 3 shows the counting chamber with preamplifier mounted on it, and Fig. 4 shows the rack containing the rest of the circuitry.

Ionization chamber. The ionization chamber used in the determination is shown in Fig. 5. The arrangement of the component parts is quite similar to that used by Fineman *et al.* (*8*).

The construction of the chamber is important in analyses that involve the determination of very small concentrations of radon. Assuming that no spurious counts are introduced by the circuitry, the limit of detection is determined by the background counting rate. This counting rate is caused by the construction materials used for the chamber, and by radon contamination of the chamber or of the filling gas. The counting rate caused by contamination of the filling gas is usually negligible in comparison to that which originates on, or in, the chamber wall and the collecting electrode.

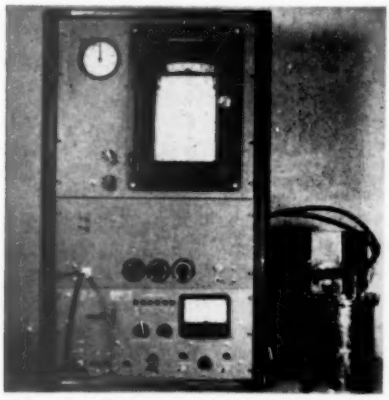

FIG. 4. Relay rack containing the scaler, main amplifier, and recorder. On the right is the ionization chamber

Fabrication of the chamber from stainless steel, pure copper, or other materials that are more free of heavy-metal impurities than is brass makes the determination of 10^{-14} gm of radium possible. The interior surfaces of chambers constructed of brass may be electroplated with copper or nickel to reduce the background rate.

The background of chambers that have been exposed to atmospheres containing radon may be reduced by evacuating the chamber to a pressure of 10^{-4} to 10^{-5} mm of Hg; heating the chamber to about 50° C during evacuation will aid the outgassing.

The chamber can quite easily be made vacuum tight; a relatively large amount of dust or other foreign material may accumulate on the insulator before high-voltage leakage across the surface is observed. The size of the insulator is such that no guard ring is necessary.

Preamplifier. The preamplifier is mounted directly on top of the chamber. The circuit used is relatively free of microphonics. Thus, no spurious counts are caused by noises or slight vibrations in the room in which the radioactivity determination is made. Connection of the preamplifier to the main amplifier is made with three plugs

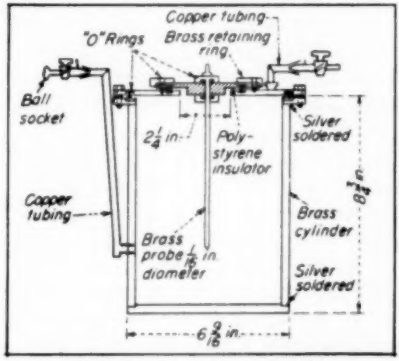

FIG. 5. Ionization chamber

mounted on the preamplifier and forming an integral part of it. The high-voltage lead is isolated from the input leads to the preamplifier, and the lead carrying the signal from the preamplifier to the main amplifier is also isolated.

The preamplifier circuit is shown in Fig. 6. A pentode-connected 6AK5 is used as the first detector stage; the signal from this stage is fed into a 6AK5 cathode-follower stage which serves to match the interconnecting coaxial cable to the main amplifier. Since the signal-to-noise ratio of the main amplifier, as well as that of the preamplifier, is good, the use of the pentode rather than the triode connection in the preamplifier, with the resultant higher gain, was warranted.

The preamplifier filament supply requires more filtering than is necessary in the main amplifier and, therefore, the preamplifier B+ is obtained from a VR-75 tube fed from the last stage of the main amplifier. Additional high-voltage filtering has also been incorporated in the preamplifier to eliminate spurious counts which may be picked up in the high-voltage cable coming from the scaler. The filtering is done with a standard *RC* filter. Coupling to the first detector stage is made through a 3,000-volt 50-$\mu\mu$f Centralab ceramic capacitor and a 1,000-megohm grid resistor.

High-gain pulse amplifier. The circuit used for the main amplifier (Fig. 7) is similar to that used by Elmore (*10*); it consists of six stages using 6AK5 tubes arranged in three feed-back loops. The basic circuit was obtained from Mr. R. Firminhac (*11*). Gain controls are placed after the first and second feed-back loops, and a short, 0.5-μsec time constant at the first gain control has been installed. The gain stages are compartmentalized to reduce noise.

The power supply for the linear amplifier and the preamplifier is mounted on the chassis with the main amplifier.

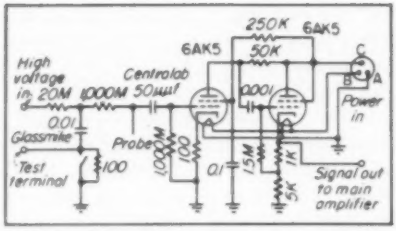

FIG. 6. Preamplifier circuit. Connection B goes to 6.3 volt d-c on power supply. For connections A and C see Fig. 7.

The filaments of the 6AK5 tubes are placed in parallel and supplied with d-c obtained from a selenium rectifier.

Since the multivibrator circuit normally furnished with commercial G-M scalers will not accept the wide range of pulse sizes observed with this counting arrangement, a Schmitt trigger circuit, fed from the last amplifier stage, has been incorporated in the amplifier. The minimum pulse height which will trigger the circuit is externally adjustable by a potentiometer that changes the bias voltage at the input grid of the trigger pair. A 6J6 cathode-follower stage has been incorporated in the amplifier to permit the signal at the last stage of the amplifier to be monitored with an oscilloscope. The tripping height of the trigger circuit is set by attaching a signal generator (pulser) to the preamplifier and setting the minimum tripping height by using the small pulse from the pulser as reference. The pulser was fabricated according to a circuit obtained from members of the Health Instruments Branch of the New York Operations Office (*12*).

When the tripping height of the circuit is set at approximately three times the half width of the hash level of the electronic circuitry, a plateau extending from about 700 to about 2,400 volts is observed. The plateau has less than a 3% slope per 100 volts. While the chambers are normally run at 2,200 volts, a lower voltage could be used.

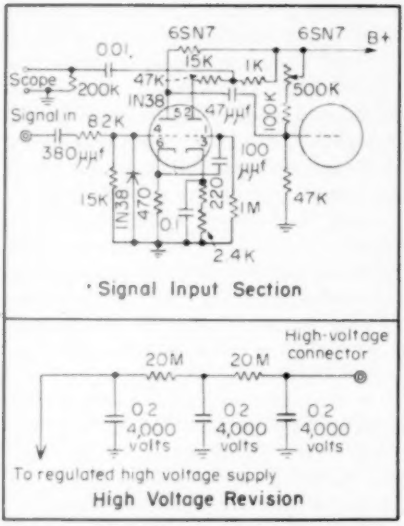

FIG. 8. Revisions made on standard commercial scaler. At the signal-in connection the Centralab capacitor, glass-mike, and high-voltage lead from the input connecter have been removed

Scaler and recorder. The scaler used to record the counts is of commercial manufacture, (Nuclear Instrument and Chemical Corp., model 163) and has a 2,500-volt power supply and scale combinations of 2, 16, 32 and 64. Since the high-voltage supply was designed for use with G-M tubes, from which large pulses are obtained, the noise filtering is insufficient for the radon application. Therefore, a three-stage *RC* filter (Fig. 8) has been mounted in the scaler. This filtering, plus that incorporated in the preamplifier, is sufficient to prevent any spurious counts from the high-voltage supply from being observed.

The Wizard register furnished with the scaler was modified (Fig. 9) so that a one milliampere Esterline-Angus recorder could be coupled to the coil. This method of recording had been used by the instrument groups of Oak Ridge National Laboratory with excellent results. The modification is the mount-

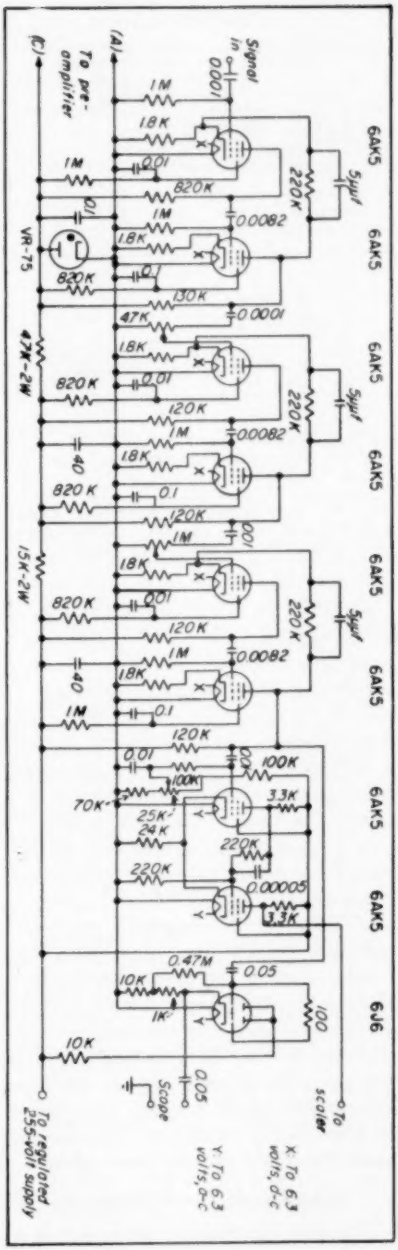

FIG. 7. Circuit for the high-gain pulse amplifier

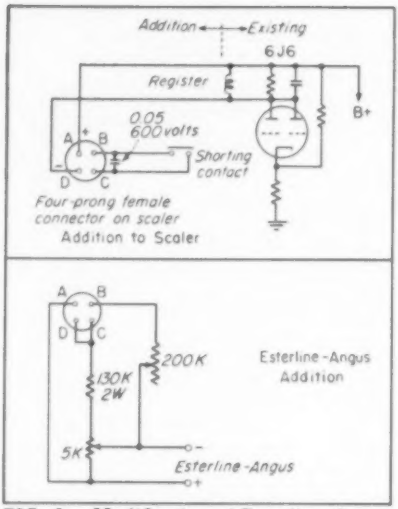

FIG. 9. Modification of Esterline-Angus connections. The shorting contact gives a long mark every ten counts

ing of a switch mechanism on the wheel of the register. This switch shorts out each time the wheel makes a complete revolution. In this way a mark is placed on the chart of the Esterline-Angus recorder each time the register is actuated; for every tenth actuation the current which is allowed to flow across the resistor network is greater and a longer line is recorded on the chart for that particular actuation. This arrangement facilitates the counting of the record, especially when fast counting rates are observed. The combination of the chart speeds available on the strip chart recorder and the scales available on the scaling unit makes the whole arrangement suitable for recording a wide range of counting rates. The chart method also makes it possible to recognize immediately gross trouble in the electronic circuits.

Standardization and Calculations

The over-all efficiency of the counting chambers and electronic circuitry was determined by comparing the counting rate with the rate theoretically obtainable from a standard of known radium content. Radium standards were obtained from the National Bureau of Standards and were certified to an accuracy of 0.3% (10^{-9}-gm standard). The precision of the radon-counting method is excellent, as shown by Table 1, and the over-all efficiency usually obtained is 66%. The known samples listed in the first column of the table were prepared by dilution of vials containing known quantities of radium that had been obtained from the National Bureau of Standards.

A counting-rate plateau exists for the setting of the gain control and the tripping height potentiometer. After initial alignment of the system a curve of counting rate vs. control setting is determined and the system adjusted for operation in the plateau region.

The deviations shown in the table are within the limits predicted from the number of random events observed. Application of the usual statistical technique (*13*) in the analysis may be used to increase the accuracy of the determination. The simple equation

$$PE = 0.67 \frac{\sqrt{N_1 + N_2}}{N_1 - N_2}$$

is useful in determining the probable error, PE, of the radon-activity determination. In this equation, N_1 is the number of counts due to radon and its decay products plus those due to the background, and N_2 is the number of counts due to background alone. Quadruplicate samples are analyzed when high accuracy is required so that the importance of errors in sampling and solution may also be minimized.

Activity calculation. The concentration of radium or radon in the original sample is calculated by using the usual exponential expressions for relating the parent daughter growth (*1*). The equations used are:

$$A_2 = \frac{A_{01}\lambda_2}{\lambda_2 - \lambda_1} (e^{-\lambda_1 t} - e^{-\lambda_2 t}) \qquad (1)$$

$$A_2 = \frac{A_{01}\lambda_2\lambda_3}{(\lambda_1 - \lambda_2)(\lambda_2 - \lambda_3)(\lambda_3 - \lambda_1)} \times [(\lambda_3 - \lambda_2)e^{-\lambda_1 t} + (\lambda_1 - \lambda_3)e^{-\lambda_2 t} + (\lambda_2 - \lambda_1)e^{-\lambda_3 t}] \quad (2)$$

In these equations, A_2 is the activity of radon after time t; A_{01} is the activity originating from the radium in solution; λ_1 is the decay constant of radium and λ_2 is the decay constant of radon. Since the value of λ_1 is infinitesimally small, the value of $e^{-\lambda_1 t}$ is, for practical purposes, unity. In practice, then, Eq. 1 becomes

$$A_2 = \frac{A_{01}\lambda_2}{\lambda_2 - \lambda_1}(1 - e^{-\lambda_2 t})$$

when used to calculate radon growth.

By using Eq. 1, the amount of radon which has been formed by allowing the flask of radium to stand for a time t may be calculated. In a strictly analogous manner, the time required for RaA to grow to equilibrium with radon after the radon has been swept into the counting chamber can be calculated by substituting the appropriate decay constants into Eq. 1. The time required for the RaB to grow to equilibrium with radon in the counting chamber may be calculated by using Eq. 2. In this equation, λ_1 is the decay constant of radon, λ_2 is the decay constant of RaA, λ_3 is the decay constant of RaB, and A_{01} is the number of alpha disintegrations from radon in a unit time. These equations result from the evaluation of a system of simple differential equations and are based upon the assumption that at zero time none of the disintegration products of the parent are present.

The time required for RaA, RaB, RaC, RaCI, and RaCII to grow into equilibrium with radon is less than four hours. The usual procedure is to allow the counting chamber to stand for four hours before the determination of the counting rate is begun. The total activity after this time will then be equal to three times the radon activity, and the radon alpha-disintegration rate can then be easily evaluated.

TABLE 1
Precision of Radon Counting Method

Radium equivalent to radon		
*Known sample** (*gm*)	*Measured* (*gm*)	*Error* (%)
1.95×10^{-7}	1.93×10^{-7}	−1.0
1.18×10^{-7}	1.20×10^{-7}	+1.7
2.89×10^{-8}	2.97×10^{-8}	+2.8
8.12×10^{-8}	7.86×10^{-8}	−3.2
1.01×10^{-11}	1.02×10^{-11}	+1.0
8.61×10^{-11}	8.73×10^{-11}	+1.4
7.40×10^{-12}	7.33×10^{-12}	−1.0
7.93×10^{-12}	7.91×10^{-12}	−0.3

* In the calculations, 2.22×10^{-12} alpha disintegrations per minute was used as the disintegration rate of radium.

* * *

The authors are indebted to J. J. Tregoning and members of the General Analytical Branch for the method of preparation and for the preparation of the solutions used in the determination.

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A Review of the Uses of Isotopic Carbon in Organic Chemical Research

Early abandonment of the attitude that isotopic carbon is still a curiosity is predicted in this review of new techniques. Organic chemists are realizing that isotopic carbon is invaluable in analytical applications and, in this role, it will surely take its place as an essential research tool

By GUS A. ROPP and O. KENTON NEVILLE

Organic Group, Chemical Division
Oak Ridge National Laboratory, Oak Ridge, Tennessee

TRACER APPLICATIONS in organic chemical research may be considered to fall into two general classes: essential uses and nonessential uses.

Essential uses involve the study of reactions, rearrangements, etc., the solution to which could be obtained in any other way only with great difficulty.

Nonessential uses involve those studies in which a tracer element facilitates the research by simplifying the procedure for the detection, identification, or analysis of one or more components of the system.

Published research has been classified in this review under various subclasses to these main divisions to demonstrate the diversity of application of the tracer tool in organic chemistry. The larger number of publications to date fall within the first, or *essential*, class. Possibly not until investigators realize the usefulness of tracers in the *nonessential* fields will the great value of isotopic techniques be realized.

Survey of Progress

The rapid increase in papers being published on the use of isotopes for organic chemical problems makes it desirable to survey progress in this rapidly expanding field at frequent intervals.

Two excellent review papers, one with respect to the study of reaction mechanisms (*1*) and the other dealing with synthetic accomplishments (*2*), which have appeared in this journal within the last year, have served well to summarize these fields. It is interesting to note, however, that since the former review appeared in June, 1950, as great a number of reports on the use of C^{13} and C^{14} in the study of organic reaction mechanisms has appeared as were summarized in that paper.

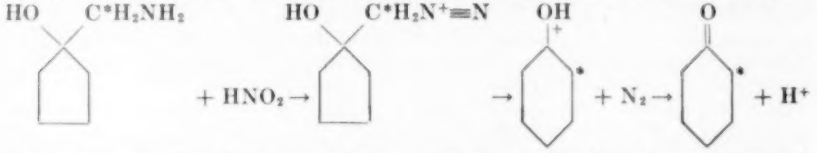
Scope of Review

The present review is restricted to a consideration of those papers involving the use of isotopic carbon as a tool in organic chemistry and will be concerned primarily with those not previously reviewed (*1*). However, so that the bibliography is more nearly complete, those papers reviewed previously (*1*) are noted by subject in the appropriate part of the classification but are not discussed, except where a paper is the sole example of an application.

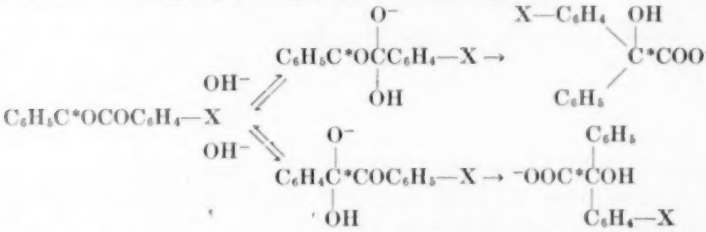
Rearrangements

Ring enlargement by Tiffeneau rearrangement. The rearrangement of 1-aminomethylcycloalkanols, on treatment with nitrous acid, to give cyclic ketones with accompanying ring expansion is an example of a Tiffeneau rearrangement and is considered to be analogous to the pinacol reaction.

Experiments with 1-aminomethyl-C^{14}-cyclopentanol and 1-amino(methyl-C^{14})-cyclohexanol have shown that rearrangement occurs such that the labeled carbon is in the 2-position of the product. The reaction probably involves an intermediate diazonium cation that rearranges spontaneously by attack of a ring methylene carbon on the external methylene position with simultaneous loss of nitrogen:



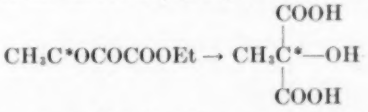
Rearrangement of α-diketones. The rearrangement of α-dicarbonyl compounds in the presence of alkali to salts of glycolic acids is a fairly general transformation. In the case of benzil, the mechanism has been indicated (*4*, *5*) as the rapid, reversible addition of hydroxyl ion at one carbonyl group, followed by the rate-controlling migration of the adjacent *R*-group. Where the two *R*-groups are dissimilar, it is possible, by labeling one carbonyl group with isotopic carbon, to determine which group undergoes preferential migration.



In a series of benzils, each having one substituted phenyl group, the migratory preferences with respect to phenyl have been found to be as follows:

p-methoxyphenyl (*6*)	0.46
p-methylphenyl (*7*)	0.63
p-chlorophenyl (*7*)	1.58
m-chlorophenyl (*7*)	4.29

A similar experiment (*7*) carried out with ethyl α,β-diketobutyrate-β-C^{14} has shown that migration of the carbethoxy group occurs exclusively:

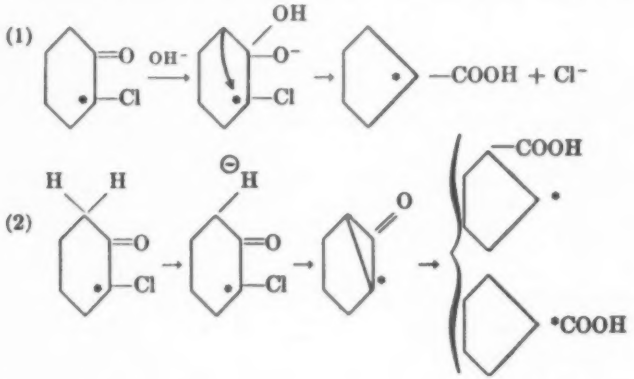


A closely related reaction, which has been postulated to occur by the intermediate formation of benzyl phenyl diketone and subsequent rearrangement, is

that of benzilideneacetophenone oxide which has been shown (*8*) to rearrange by benzyl group migration exclusively:

$$C_6H_5C^*OCHCHC_6H_5 \rightarrow [C_6H_5C^*OCOCH_2C_6H_5] \rightarrow C_6H_5C^*(CH_2C_6H_5)(OH)(COOH)$$

Favorski reaction. The Favorski reaction consists of the rearrangement of an α-haloketone in alkaline solution to an α,α-disubstituted acid by migration of one *R*-group. One mechanism that has been written for this reaction is analogous to that for the benzilic acid rearrangement. A second involves a symmetrical cyclopropanone intermediate. The reaction has now been studied (*9*) using α-chlorocyclohexanone, labeled with carbon-14 at the chlorine-substituted carbon atom. With this compound, a benzilic acid type of rearrangement would lead to cyclopentanecarboxylic acid labeled exclusively in the α position. The cyclopropanone route would result in equal amounts of the acid labeled in the α and β positions:



Degradation of the cyclopentanecarboxylic acid revealed equal labeling in the α and β positions, thus eliminating the first mechanism and lending support to the route involving a cyclopropanone intermediate.

Rearrangements of carbon atoms in *t*-butyl and *t*-amyl derivatives. Several carbonium ion reactions of C^{14}-labeled *t*-butyl and *t*-amyl derivatives have been investigated to determine the ease of rearrangement of these compounds to substances with identical carbon skeletons but different locations of the isotopic atoms. Such studies are of importance in the clarification of reactions involving carbonium ions since they enable determination of the magnitude of the interconversion energy barriers free of complications resulting from differences in energy between starting materials and end products.

$$\underset{\text{I}}{(CH_3)_3C^*—X} \rightleftharpoons \underset{\text{II}}{(CH_3)_2(C^*H_3)C—X}$$

$$\underset{\text{III}}{(CH_3)_2C^*XCH_2CH_3} \rightleftharpoons \underset{\text{IV}}{CH_3(C^*H_3)CXCH_2CH_3} \rightleftharpoons$$

$$\underset{\text{V}}{(CH_3)_2CXC^*H_2CH_3} \rightleftharpoons \underset{\text{VI}}{(CH_3)_2CXCH_2C^*H_3}$$

The starting compounds for the rearrangement studies were 2-methyl-2-propanol-2-C^{14} (**I**, X = OH), 2-methyl-2-butanol-2-C^{14} (**III**, X = OH), and 2-methyl-2-butanol-1-C^{14} (**IV**, X = OH).

In the first reaction studied, that of *t*-butyl alcohol (**I**, X = OH) with hydrochloric acid to give *t*-butyl chloride, no rearrangement to **II** (X = Cl) or the corresponding alkene could be detected. Similarly, *t*-amyl alcohol (**IV**, X = OH) gave no rearrangement in the conversion to the respective chloride.

The second reaction type studied was the conversion of the alcohols to alkenes by dehydration with sulfuric acid. Although this reaction frequently leads to rearranged products, the dehydration of **III** (X = OH) and **IV** (X = OH) gave trimethylethylene with less than 2% change in the isotopic distributions of carbon chains in 18*N* sulfuric acid at 140° C. It is apparent from these results and those with hydrochloric acid that rearrangements of the type **I** $\rightleftharpoons$ **II** and **III** $\rightleftharpoons$ **IV** do not seem to be important in the usual essentially nonreversible metathetical carbonium ion reactions of *t*-butyl and *t*-amyl derivatives.

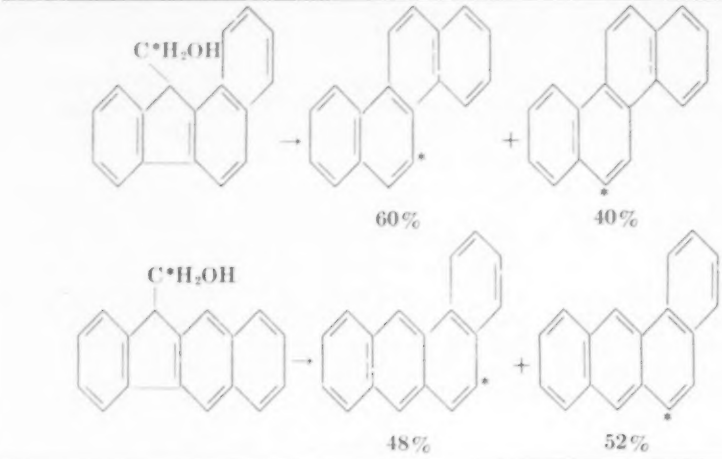
The third reaction studied was that of tertiary halides with water-promoted aluminum chloride, the most severe of the reaction conditions. Treatment of 2-methyl-2-chlorobutane-1-C^{14} (**IV**, X = Cl) with aluminum chloride at 0° C gave 20% recovered *t*-amyl chloride whose isotpic analysis showed almost complete equilibration between C-1 and C-4. There was little or no radioactivity at C-2 or C-3. Treatment of 2-methyl-2-chlorobutane-2-C^{14} (**III**, X = Cl) with aluminum chloride under conditions allowing 57% recovery of chloride gave a partially rearranged product with about 70% of the possible exchange between C-2 and C-3 and weak radioactivity at C-1 and C-4. These results, as well as those obtained with 2-methyl-2-chlorobutane-1-C^{14}, demonstrate that little exchange occurs between the methyl groups and the carbons in the center of the chain during the isotopic equilibration between C-2 and C-3 or between C-1 and C-4.

The aluminum-chloride-catalyzed rearrangement of 2-methyl-2-chloropropane-2-C^{14} (**I**, X = Cl), which occurred much less readily, gave 48% of recovered chloride containing only 1–3% of 2-methyl-2-chloropropane-1-C^{14} (**II**, X = Cl).

The selective rearrangements above, which are interpretable on the basis of carbonium ion intermediates, contrast strongly with those reported (*11*, *12*) on the gas-phase isomerization of C^{13}-labeled butanes over moisture-promoted aluminum bromide in which a deep-seated mixing of carbon atoms was found to occur, which could not be explained by a Whitmore mechanism.

Wagner rearrangements of aromatic hydrocarbons. The dehydration of 9-fluorenylcarbinol by phosphorus pentoxide gives, by a Wagner rearrangement, phenanthrene. Extension of this reaction to benzo-substituted C^{14}-labeled fluorenylcarbinols has led to the synthesis of chrysene-5,6-C^{14}_1 and benz(*a*)anthracene-5,6-C^{14}_1. Since the fluorenylcarbinols and corresponding carbonium ions are unsymmetrical, unequal C^{14}-labeling in the 5 and 6 positions might be expected to occur. Degradation and C^{14} assay has shown the chrysene (*13*) to consist of 60% of 5-labeled and 40% of 6-labeled compound. The benz(*a*)anthracene-5,6-C^{14}_1 (*14*) was labeled 48% in the 5 position and 52% in the 6 position. This is illustrated at the top of the following page.

Willgerodt reaction. An anomaly that has existed in the results of tracer studies in the Willgerodt reaction has now been removed. Previous results (*14*) had shown



that when C^{14}-carbonyl-labeled acetophenone was treated with yellow ammonium sulfide, the phenylacetamide formed contained only 3% of the radioactivity in the carbonyl group, the remainder being in the α position.

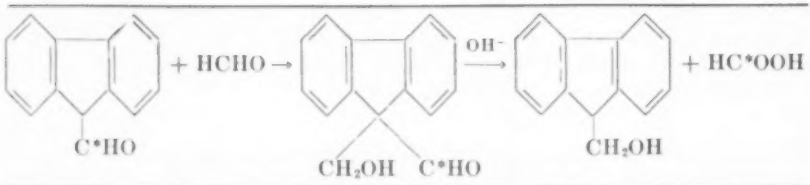
The phenylacetic acid, found in small amounts as a byproduct, was found to be labeled to the extent of 75% in the carboxyl group. These results led to the interpretation that the phenylacetamide and the phenylacetic acid are formed by different paths, the latter by some mechanism involving rearrangement of the carbon chain.

Recent studies (*16*) with carbonyl- and methyl-labeled acetophenone by other workers have indicated no skeletal rearrangement in the formation of either product. It was shown that thermal decarboxylations of phenylacetic acid for isotopic analysis are unreliable.

Other literature include papers on the reaction of phenylglyoxal with alkali (*17, 18*); the mechanism of the Arndt-Eistert reaction (*19*); the Willgerodt reaction, Kindler modification (*20*); and the isomerization of hydrocarbons (*11, 12*).

Reactions Involving Loss or Gain of Carbon Fragments

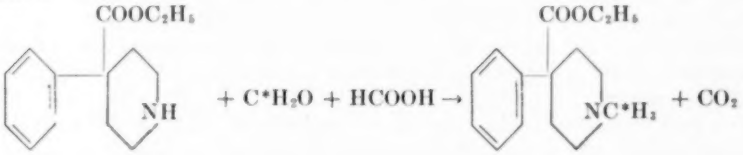
Reaction of 9-formylfluorene with formaldehyde. A particularly interesting reaction recently described is that of aqueous alkaline formaldehyde with 9-formyl-



fluorene. This aldehyde, which, if unlabeled, appears to undergo a normal crossed Cannizzaro reaction to give 9-fluorenylcarbinol, is found (*21*) to react by displacement of the aldehyde group. The products of the reaction of unlabeled formaldehyde with 9-(formyl-C^{14})-fluorene are nonradioactive 9-fluorenylcarbinol and formic-C^{14} acid. Of the possible mechanisms suggested (*22*), the one diagrammed at the bottom of the preceding page is therefore supported.

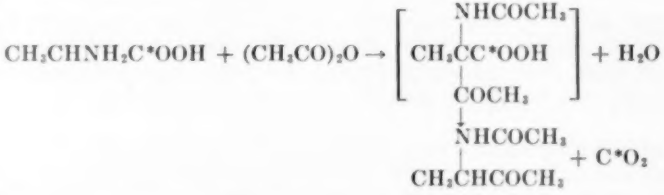
No exchange was found between formaldehyde and sodium formate.

Source of the N-methyl group in the preparation of Demerol. The source of the N-methyl group in amine methylations using a mixture of formaldehyde and formic acid has been shown (*23*) to be the formaldehyde in the preparation of demerol-C^{14}; the formic acid acts only as a reducing agent. When the alkylation of 4-phenyl-4-carbethoxypiperidine was carried out using formaldehyde-C^{14} and inactive formic acid, the resulting demerol was radioactive, its specific activity being equal to that of the formaldehyde. When the experiment was carried out with formic-C^{14} acid and nonradioactive formaldehyde, the specific activity of the carbon dioxide was identical with that of the formic acid, the demerol being inactive.



Formation of α-acylaminoketones from amino acids. The mechanism of the conversion of α-amino acids to α-acylaminoketones and carbon dioxide by means of acetic anhydride in the presence of a base has been pictured as one of two processes: (1) condensation of one of the methyl groups of the acetic anhydride with the carboxyl group of the amino acid, solvolysis of the resulting mixed anhydride and decarboxylation of the β-keto-acid, or (2) the acetylation of the α position of the amino acid, preceded or followed by loss of carbon dioxide from its carboxyl group.

With alanine-1-C^{14} and phenylalanine-1-C^{14} used with nonradioactive acetic anhydride, the carbon dioxide evolved in the reaction was found (*24*) to have the same specific activity as the carboxyl group of the amino acid used. The 1-phenyl-2-acetamino-3-butanone and 3-acetamino-2-butanone obtained from the respective amino acids were found to be nonradioactive. The probable reaction mechanism is then:



Preparation of potassium cyanate from urea. In connection with the problem of preparing urea labeled with both C^{14} and N^{15}, it has been found (*25*) that fusion of potassium carbonate with urea gives potassium cyanate containing carbon derived

only from the urea. The accompanying carbon dioxide results from the potassium carbonate:

$$K_2CO_3 + 2NH_2C^*ONH_2 \rightarrow 2KC^*NO + CO_2 + H_2O + 2NH_3$$

Decomposition of diacetyl peroxide in acetic acid. The thermal decomposition of diacetyl peroxide in acetic acid has been postulated (*26*) to give the observed products by a free radical chain reaction:

$$(CH_3COO)_2 \xrightarrow{\Delta} CO_2 + CH_3\cdot + CH_3COO\cdot$$
$$CH_3\cdot + CH_3COOH \rightarrow CH_4 + \cdot CH_2COOH$$
$$2\cdot CH_2COOH \rightarrow HOOCCH_2CH_2COOH$$
$$2CH_3COO\cdot \rightarrow CH_3COOCH_3 + CO_2$$
$$(CH_3COO)_2 + CH_2COO\cdot \rightarrow CH_3COOCH_3 + CO_2$$

The use of acetic-2-C^{14} acid and unlabeled diacetyl peroxide should yield, by the path shown above, unlabeled carbon dioxide, methane, and methyl acetate plus labeled succinic acid in which each methylene group should have the same specific activity as the methyl group of the acetic acid. From the data (*27*) obtained by degradation of the products of this reaction, the mechanism above has, in general, been confirmed. The only anomaly found was in the presence and distribution of radioactivity in the methyl acetate; this could not be explained on the basis of the mechanism above.

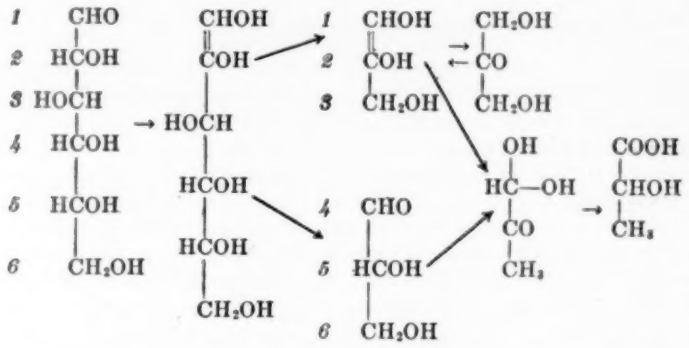
As a part of the research, it was demonstrated that exchange between acetic acid and diacetyl peroxide or between acetic acid and methyl acetate did not occur under the conditions of the experiment.

Chemical formation of lactic acid from glucose. The conversion of glucose in alkaline solution to lactic acid has been postulated to involve the intermediate formation of D-glucose-1,2-enediol which breaks between carbons 3 and 4 to form one molecule each of glyceraldehyde and triose enediol. The glycerol was postulated to be readily convertible to triose enediol and thence to dihydroxyacetone. Pyruvaldehyde was the immediate precursor of lactic acid. By this mechanism, the aldehyde carbon of glucose would become the carboxyl- and methyl-carbon of lactic acid, due to randomization of this carbon atom as it passed through symmetrical dihydroxyacetone. The equilibration of carbons 1 and 3 of glucose was substantiated (*28*) by isolation and degradation of the lactic acid formed from D-glucose-1-C^{14}.

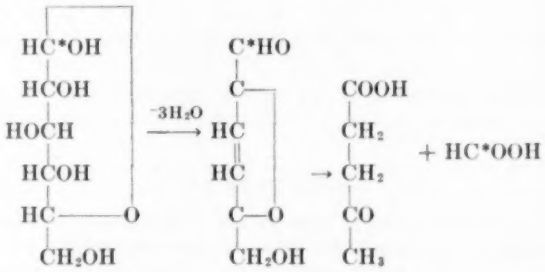
When glucose-3,4-C^{14}_2 was similarly treated, randomization of carbons 4 and 6 was shown not to occur, thus eliminating the postulate involving rearrangement of glyceraldehyde to dihydroxyacetone. The mechanism for the formation of lactic acid was therefore written as shown at the top of the next page.

From the data, the conclusion was drawn that more triose enediol was converted to lactic acid than was glyceraldehyde.

Action of hydrobromic acid on D-glucose-1-C^{14}. D-glucose, on treatment with strong mineral acids, undergoes dehydration and cleavage to give, as final products, levulinic and formic acids. 5-Hydroxymethyl-2-furaldehyde, postulated as an intermediate in the reaction, would yield formic acid by cleavage between C-1 and C-2 of glucose. D-glucose-1-C^{14} when treated with hydrobromic acid has been shown (*29*) to give nonradioactive levulinic acid and formic-C^{14} acid of

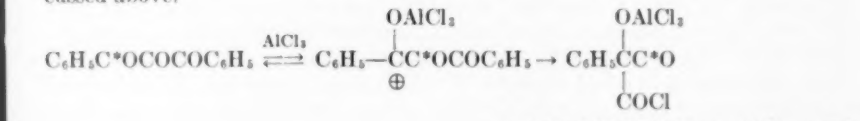


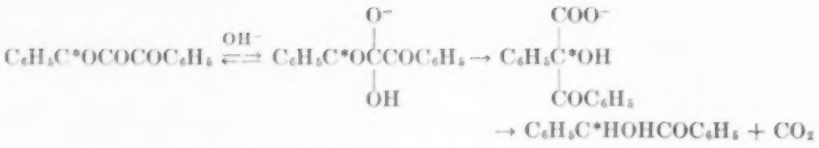
specific activity identical with that of glucose, thus substantiating the mechanism proposed.



Decarbonylation of diphenyltriketone. Diphenyltriketone, like a number of other α-polycarbonyl compounds, loses carbon monoxide or dioxide under the influence of a variety of catalysts, both acid and basic, to given benzil or benzoin. This interesting transformation has now been studied (*6*) with C^{14} labeling to determine whether the center or side carbonyl group is lost under three catalytic conditions. With aluminum chloride, cupric acetate, and sodium hydroxide, the center carbonyl group was lost in each case. The products formed were, respectively, benzil and carbon monoxide, benzil and carbon dioxide, and benzoin and carbon dioxide. Aluminum chloride catalysis in the presence of water produced benzoin and carbon dioxide.

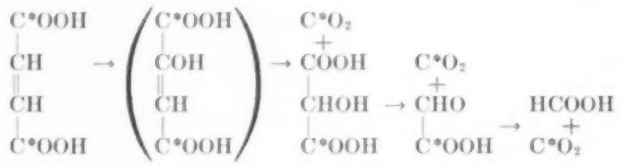
Exactly analogous results were obtained with *p*-methoxyphenyl phenyl triketone. The results are in accord with a formulation involving a Wagner-Meerwein transformation in the aluminum chloride and cupric-acetate-catalyzed reactions. The base-catalyzed conversion is analogous to the benzilic acid rearrangement discussed above.





Acid oxidation of fumaric acid. An early study (*30*), using C^{11}, which has not been reviewed (*1*) is that of the oxidation of fumaric acid in acid solution to give carbon dioxide and formic acid. Oxidation of C^{11}-carboxyl-labeled fumaric acid with potassium permanganate in 1.5*N* sulfuric acid at 35–50° was found to give carbon dioxide containing all of the radioactivity plus inactive formic acid.

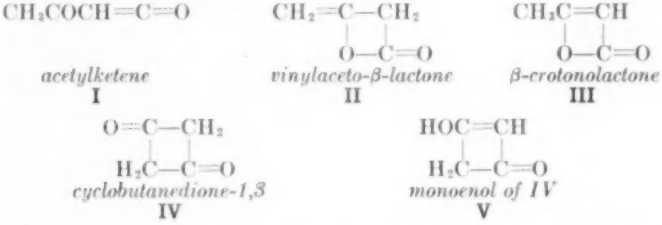
Since the formic acid must have been derived from a methine position of the succinic acid, the authors have proposed the following sequence as the reaction path:



Other studies have resulted in papers on fatty-acid oxidation (*31–34*); oxidation of propene (*35*); decarbonylation of ethyl pyruvate (*36*); and synthesis of uvitic acid (*37*).

Structure Proof

Only one example of the use of isotopic methods in structure proof has been reported in organic chemical studies: that of the determination of the structure of ketene dimers (*38*). Five structures have been postulated for diketene, only one (**IV**) of which has two equivalent carbonyl groups:



acetylketene **I**

vinylaceto-β-lactone **II**

β-crotonolactone **III**

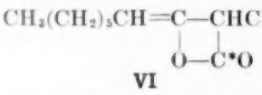
cyclobutanedione-1,3 **IV**

monoenol of IV **V**

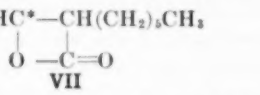
Since higher aldoketene dimers have chemical and physical properties analogous to those of diketene and probably have analogous structures, the problem was attacked by studying the mixed dimer prepared from methyl and hexyl ketenes. The methyl ketene was prepared from propionyl-1-C^{14} chloride, and the hexyl ketene was inactive. If the mixed dimer has a structure analogous to **IV** or **V** in which there are two equivalent or nearly equivalent carbonyl groups, alcoholysis of the dimer, followed by treatment with sodium ethoxide would give a mixture of ethyl propionate and ethyl caproate in which the C^{14} was equally distributed between the two esters. Any contribution in the mixed dimer of a form analogous to **IV** or **V** would lead to a corresponding amount of labeled ethyl caproate. The

radioactivity found in the ethyl caproate indicated the maximum amount of structure of type **IV** or **V** to be 3%.

The mixed dimer, shown to have little of the type **IV** or **V** structure, could exist in two forms, depending on which ketene furnished the C=C=O group of a structure similar to **I** or the carbonyl groups of the lactonic structures similar to **II** or **III**. From radioactive analysis of the carbon dioxide formed by decarboxylation of the acetoacetic acids resulting from the hydrolysis of the mixed acetoacetic esters, it was found that 67% had the structure **VI** and 33% the structure **VII**.



VI



VII

From the radioactivity data, together with results of infrared and Raman measurements and comparison of physical properties, the authors suggest structures **II** and **III** as being most likely for diketene.

Isotope Effect

Significance in tracer research. Since a fundamental assumption in isotopic-tracer technique is that isotopic isomers (labeled and unlabeled molecules of the same structure) are indistinguishable chemically, it has been of importance to determine whether there is a measurable difference in the rates of reaction of C^{13}- or C^{14}-labeled and unlabeled compounds.

In the study of a number of reaction systems, workers have reported differences of the order of 3–5% for C^{13}-labeled compounds and of 8–40% for C^{14} reactants. Since the methods used have varied both in type and precision, a quantitative correlation or interpretation of the results is difficult at present. Following is a list of the reactions in which measurable isotope effects have been reported:

(*a*) dissociation of propane-1-C^{13} by electron impact (*11*)
(*b*) thermal dissociation of propane-1-C^{13} (*39*)
(*c*) thermal decarboxylation of malonic-1-C^{14} acid (*40*)
(*d*) thermal decarboxylation of bromomalonic-1-C^{14} acid (*40*)
(*e*) thermal decarboxylation of malonic-1-C^{13} acid (*41*)
(*f*) alkaline hydrolysis of C^{14}-carbonyl-labeled ethyl benzoate (*42*)
(*g*) rearrangement of C^{14}-carbonyl-labeled benzil (*43*)
(*h*) urease catalyzed hydrolysis of urea-C^{14} (*44*)
(*i*) thermal decomposition of oxalic acid of normal C^{13} enrichment (*45*)
(*j*) sulfuric acid dehydration of formic-C^{14} acid (*46*).

In all but one of the reactions above, the reaction rate of the isotopically substituted molecules was lower than that for the C^{12} species. In the hydrolysis of urea with urease, the first fraction of carbon dioxide released was enriched in carbon-C^{14} dioxide, thus indicating a higher reaction rate for the C^{14} molecules.

In the following reactions, isotope effects have been sought, and found absent or negligibly small:

(*a*) Cannizzaro reaction of C^{14}-carbonyl-labeled benzaldehyde (*43*)
(*b*) benzoin condensation of C^{14}-carbonyl-labeled benzaldehyde (*43*)
(*c*) pyrolysis of barium adipate of normal C^{13} enrichment (*47*).

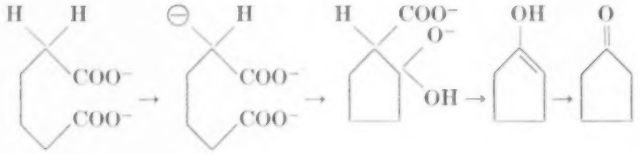
The large differences in the reaction rates of labeled and unlabeled molecules indicate the dangers in quantitative interpretation of data from isotopic carbon experiments. Since, if present, an isotope effect will not be observed in those

cases where the reaction involving the labeled position goes to completion, the interpretation of such data should be unaffected.

Where the reaction in question involves a choice between a labeled and unlabeled position, where the labeled molecule may react to form either of two or more different reaction products, or where the conversion yield of a labeled reactant is low, the data obtained in tracer work may be in error due to an isotope effect.

An example of the effect of such considerations on tracer experiments is in the measurement of migratory preferences in the substituted benzils mentioned previously. Since an isotope effect of 11% has been demonstrated in the rearrangement of unsubstituted benzil, it seems probable that superimposed on the results of the effect of substituents on the direction of migration is a second effect caused by the difference in reactivity of C^{12} and C^{14} bonds. In this case, the isotope effect can be determined by comparison of the results above with those obtained with benzils similarly substituted but labeled in the other carbonyl group.

Use as a research tool. Since, from theoretical considerations, it seems possible that an isotope effect will be observed only if the labeled atom is affected in the rate-determining step, the suggestion has been made (*46*) that determination of the existence or absence of an isotope effect in a chemical reaction gives an insight into the mechanism of chemical processes. In this way, the lack of an isotope effect in the pyrolysis of barium adipate has been interpreted as evidence that the decarboxylation step is not rate-controlling. It is suggested, therefore, that removal of a proton in the α position is more likely to be the slow step in the reaction:



Equilibrium studies. It has usually been considered that the equilibrium constant, K^*/K, in a C^{14}-C^{12} isotopic exchange reaction (a reversible process) would deviate only slightly from unity, while in irreversible reactions the ratio, k^*/k, of the rate constants for the reaction of the labeled compound to that for the reaction of the unlabeled compound might be different from unity by 10% or more. Very recently, however, it has been reported (*48*) that, in a C^{14}-C^{12} isotope exchange reaction involving labeled carbonate, the equilibrium constant was found to deviate 11% from unity. The equilibrium involved is:

$$[Co(NH_3)_4C^{12}O_3]^+ + C^{14}O_3^{=} \rightleftarrows [Co(NH_3)_4C^{14}O_3]^+ + C^{12}O_3^{=}$$

and the equilibrium constant,

$$K = \frac{[[Co(NH_3)_4C^{14}O_3]^+]}{[[Co(NH_3)_4C^{12}O_3]^+]} \Big/ \frac{[C^{14}O_3^{=}]}{[C^{12}O_3^{=}]} = 0.89$$

was evaluated by comparison at equilibrium of the specific activity of ionic carbonate (counted after precipitation with barium ions) with the specific activity of the complexed carbonate (counted after acid decomposition of the complex and precipitation of the released carbon dioxide with barium ions).

Exchange Reactions

The study of isotopic exchanges has not been exploited with the carbon isotopes to the same extent as with other elements. In general, such reactions have been studied incidentally, in connection with other work. Examples mentioned above are the lack of exchange between formaldehyde and formic acid in the preparation of demerol and in the reaction of 9-formylfluorene with formaldehyde, and the lack of exchange of acetic acid with methyl acetate or with diacetyl peroxide in connection with the study of the decomposition of diacetyl peroxide.

Sodium formate-sodium carbonate exchange. In view of the lack of exchange between formate and formaldehyde in basic solution, a recent paper (*49*) is of interest which reports nearly complete exchange between sodium formate and sodium carbonate in water solution at 250°. When a water solution of equal weights of sodium carbonate-C^{14} and inactive sodium formate was heated in a Pyrex tube at 250° for 4 hours, 30% of the activity was found in the sodium formate. A similar experiment with unlabeled sodium carbonate and C^{13}-labeled formate gave analogous results.

An experiment under similar conditions with labeled sodium acetate and unlabeled sodium carbonate revealed no exchange. The authors have suggested that a conceivable mechanism involves equilibrium with oxalate ion.

Acetic anhydride-sodium acetate exchange. In connection with the preparation of C^{11}-labeled unsaturated acids, it was found (*50*) that the exchange between acetic anhydride and sodium acetate-1-C^{11} was surprisingly rapid at room temperature. Within 20 minutes, 55% of the theoretical exchange had occurred, regardless of the low solubility of solid sodium acetate in acetic anhydride. Similar results were obtained with sodium butyrate and acetic anhydride.

Exchange reactions previously reviewed include the exchange of the formamido group (*51*).

Nonessential Uses of Isotopic Carbon

Dilution Method Analysis

Although the theoretical basis for isotope dilution analysis was laid (*52*) in 1934, and the method has been well discussed (*53*) and tested with carbon isotopes in biological fields (*54–56*), this important analytical tool has had little use in organic chemistry. The quantitative analysis for one or more organic components in a reaction mixture is frequently difficult if the compounds are similar, since purity and quantitative recovery are mutually exclusive. With isotope dilution methods, the quantitative isolation of a compound is not necessary; all that is needed is sufficient pure sample for isotopic assay. Since several variations of the method are known, the technique is quite flexible and can be adapted to the analysis of most compounds for which a labeled form is available. In the discussion of the various methods below, a radioactive tracer, such as carbon-14, is assumed

for simplicity of presentation, but certain stable isotopes such as carbon-13 are also useful in isotope dilution technique.

Isotope addition method. Analysis for a macro amount, x, of an organic compound may be accomplished by the addition of a known weight, a, of this same component labeled in any position. If the known total radioactivity added is A, then the initial specific activity, S_0, is A/a. After mixture and equilibration, a pure sample of the compound is isolated, purified, and assayed for radioactivity. The new, lower specific activity, S, will be equal to $A/(a + x)$. The grams of material originally present, x, is then calculated from the relationship:

$$\frac{S_0}{S} = \frac{A/a}{A/(a+x)} = \frac{(a+x)}{a}$$

Therefore: $x = \frac{aS_0 - aS}{S}$.

The precision of the method is dependent on the ratio of the values x and a; the larger the ratio x/a the less is the effect on the calculated value of x of errors in the radioanalyses.

An excellent demonstration of the method has been accomplished (*57*) with sulfur-35 as the labeling atom in the quantitative analysis of a three-component mixture of dibenzyl-sulfide, -sulfoxide and -sulfone. Data are given that indicate an average error of less than 1%.

Labeled-reagent addition method. A modification of the method just noted above has been described (*58*). The mixture is treated with a labeled reagent which forms, quantitatively, a stable derivative of the desired component. The quantity of the compound may be calculated from considerations similar to those above from radioanalysis of the recovered derivative.

Carrier addition method. When the analysis desired is for one or more radioactive components of a system, a known amount, G, of the unlabeled component (carrier) desired is added and allowed to equilibrate with its like radioactive compound of known specific activity, S_0. If the material is now separated and purified from all other radioactive constituents, its radioassay will indicate a new specific activity S. The amount of the radioactive compound originally present, y, can then be determined from the following equations:

$$\frac{S_0}{S} = \frac{(G+y)}{y}$$

Therefore: $y = \frac{SG}{S_0 - S}$

The utility of this method has been demonstrated in the yield determinations of intermediate and final products in a series of organic reactions devoted to the synthesis of diethyl malonate-2-C^{14} of high specific activity (*59*).

In these reactions, the scale was so small that the isolation and identification of the intermediate products was nearly impossible. At each stage of the reaction sequence, a small aliquot of the reaction mixture was diluted with a large known amount of the appropriate pure unlabeled compound. Radioactive assays of the purified samples permitted determination of the yield of each step.

Double dilution method. In the method just discussed above, it is required that the specific activity of the sought component be known by calculation from the radioassay of a "parent" or "daughter" compound. If the compound to be analysed arises by some process such that its specific activity cannot be determined, the technique of double dilution may be used to determine its specific activity and quantity simultaneously (*60*).

If two aliquots of the desired radioactive component are diluted with

different amounts of inactive carrier, the samples of isolated material will have different specific activities. If S_0 is the initial specific activity and S_1 and S_2 are the specific activities of the two diluted aliquots, the following equations will apply:

$$S_1 (G_1 + z) = S_0 z,$$
$$S_2 (G_2 + z) = S_0 z,$$

where G_1 and G_2 are the amounts of carrier added to the two aliquots, and z is the amount of radioactive component sought.

Solving the two equations, first for z, and then for S_0, the equations below are obtained:

$$z = \frac{S_2 G_2 - S_1 G_1}{S_1 - S_2}$$

and

$$S_0 = \frac{S_1 S_2 (G_1 - G_2)}{S_2 G_2 - S_1 G_1}$$

Radioassay to determine S_1 and S_2 will allow the calculation of z and S_0, the weight and specific activity, respectively, of the desired component. Although the method is inherently much less precise than those discussed above, the precision in calculation is increased by making G_1 and G_2 as different as possible.

A recently reported test (*61*) of the method in an organic system has indicated its utility and limits of precision. During a synthesis of chrysene-5,6-C^{14}, an intermediate product was obtained that resisted purification. Two successive dilutions were made so that a sample from each dilution could then be more easily purified. From the radioassays of the two samples, the calculated specific activity was found to be 586 μc/mmole as compared with the true specific activity of 595 μc/mmole.

Analysis of optical isomers. When a reaction involves a radioactive compound containing optical isomers, the amount of each isomer can be determined by the isotope dilution method. By addition of either D or L form of the nonradioactive material, formation of a derivative by reaction with one form of a second optically active compound, and isolation and radiochemical analysis of the pure diastereoisomeric derivative, the percentage of either D or L form present may be calculated by methods analogous to those described above.

By this method, it was demonstrated that glutamic acid isolated from a physiological system was free of D-glutamic acid (*62*).

Use of stable isotopes. Although the examples above have been given in terms of a radioisotope, identical techniques are possible with stable isotopes. In calculation, the atom per cent of the rarer isotope is used in place of the specific activity. When the enrichment of the isotope is greater than one atom per cent, an additional complication is introduced by the necessity of using a corrected molecular weight. An excellent theoretical treatment of the general case of isotope dilution with stable isotopes has been written (*63*).

Use of isotope dilution method in theoretical organic chemistry. An example of the use of an isotope dilution method of analysis in the study of a problem in theoretical organic chemistry is in the attempt to find crossed products in the benzidine rearrangement (*64*). Although crossed products have never been isolated by chemical methods in the study of the rearrangements of a mixture of two dissimilar hydrazobenzene derivatives, the possibility exists that the methods used lacked the proper sensitivity.

A preliminary attack on the problem was made by studying the rearrangement of the unsymmetrical hydrazobenzene, 2-(methyl-C^{14})-2′-ethoxyhydrazobenzene, to the semidine. An

equal amount of nonradioactive o-tolidine dihydrochloride, a possible crossed product, was added and the mixture was separated. On careful purification, the isolated derivative of the added component contained negligible radioactivity thus eliminating the possibility of the formation of significant amounts of this crossed product in the rearrangement.

Other Analytical Uses

Demonstration of purity. When the interpretation of data from the study of an organic reaction depends upon the purity of some component, isotopic techniques are frequently of value. (In the discussion below, a radioactive isotope is assumed, although the methods, with the exception of autoradiographic techniques, are equally applicable for stable isotopes.)

One method consists of equilibrating with the nonradioactive component an appropriate (though not necessarily known) amount of radioactivity in the form of this labeled compound. When, after repeated purifications, such as derivative preparation or crystallization, the specific activity of the product becomes constant, this may be accepted as evidence of the purity of the compound.

The reverse of this method, in which the component to be purified is radioactive, consists of the addition of pure carrier and purification to constant specific activity (*65*).

Determining the purity of a radioactive component that may be contaminated with other radioactive materials may be accomplished by the use of paper chromotography and the autoradiographic technique. This method has been of importance in biological research (*66*).

In the successive preparation of several radioactive compounds, one from the other, the radioactivities per mole of each compound should be identical with every other one of the series (*18*), if there has been no dilution of any compound. This criterion of purity is frequently of value in the study of organic reactions in which the purity of one member of a series may be in doubt.

The reverse of this technique is of value when a compound of unknown structure is derived in such a reaction series. If such a compound is formed by incorporation of only one labeled position into the molecule, the determination of the specific activity gives, by calculation, the molecular weight. If the compound formed occurs by dimerization or by incorporation of two or more labeled positions, the "radioactive equivalent" thus calculated, together with a molecular weight determination, gives a valuable clue to the identity of the compound.

Study of gaseous systems. Though the great importance of isotopic carbon in the study of gaseous systems is yet to be realized, preliminary results in the study of the Fischer-Tropsch process (*67*, *68*) and in the oxidation of acetylene (*69*) have demonstrated its utility both as tracer and as an aid to analysis. These papers, however, are more closely related to industrial or physical chemistry than to organic chemistry and will not be reviewed here.

Determination of physical properties. Although the applicability of isotopic-carbon techniques as an analytical aid in the determination of physical properties such as vapor pressures, solubilities, equilibrium constants, etc., is obvious, little of such research has been reported.

One report, which will be of interest to organic chemists, is that of the determination of distillation data in the ternary system, methanol, ethanol and water, in which methanol-C^{14} was used to facilitate analysis (*70*). As the authors point out, the difficulty and labor involved in determining data in multicomponent distillations has retarded the design theory of distillation equipment. The use of a radioactive component reduces the difficulties tremendously.

Such techniques to determine column efficiencies should be of real value in laboratory work.

* * *

The use of isotopic-carbon techniques in organic chemical research is fast growing out of its infancy. Despite this, the heavy emphasis on research into rearrangements and carbon eliminations, when compared to the lighter emphasis on analytical applications, indicates that isotopic-carbon methods are still looked upon as a curiosity rather than as a research tool. The prediction is inescapable, however, that isotopic carbon will in the years ahead, as with other techniques that have become vital to research, become an essential part of organic research.

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(*Continued on page 70*)

Radioisotope Procedures with Farm Animals—II*

QUANTITATIVE ADMINISTRATION and BLOOD-SAMPLING

Supplementing the metabolism facilities for cattle, sheep, and swine reviewed in Part I of this paper are the methods described below for taking blood samples and administering radioisotopes intravenously, intraperitoneally, and orally

By SAM L. HANSARD, C. L. COMAR, and M. P. PLUMLEE

University of Tennessee – Atomic Energy Commission Agricultural Research Program
Oak Ridge, Tennessee

As a direct consequence of nuclear energy developments, there has been an increasing use of large animals for a variety of studies involving radiation, radioisotopes and metabolism investigations (*1-10*). When farm animals are used, many problems are introduced that are new to both the research worker accustomed to handling these animals, and to specialists in related fields.

Part I of this paper (*11*) described facilities and methods for controlled animal management based on adapted metabolism units for complete immobilization, and subsequent restriction to a limited area, for quantitative administration of activity and separate collection of excreta during the experimental period. This part of the paper describes methods and procedures suitable for the quantitative administration of radioactivity to the animal and for the collection of frequent blood samples.

General Considerations

The usual precautions to be observed in the handling of radioisotopes have been well documented in the literature (*12-17*).

External radiation hazards are minimized by using shielding barriers properly, maintaining exposure time at a minimum, providing for maximum distance between the worker and the activity, and using only that amount of activity necessary to accomplish the objective of the experiment.

The likelihood of radioactive materials' entering the body is minimized by the use of rubber gloves and protective clothing, maintenance of sanitation and good housekeeping procedures, and avoidance of contamination in the form

FIG. 1. Demonstrating quantitative intravenous administration of a radioisotope to an immobilized pig; with this small animal the needle is inserted directly into the vein. Note Lucite syringe shield for beta emitters

* Published with approval of the Director of the Tennessee Agricultural Experiment Station. See item *11* in the bibliography on page 45 for reference to Part I of this paper.

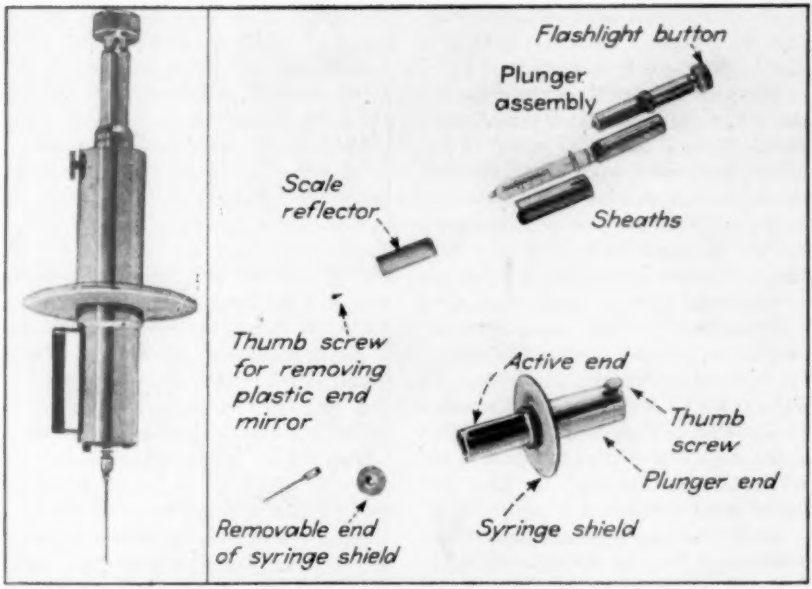

FIG. 2. A syringe shield suitable for gamma emitters

of radioactive gases, vapors, or dust particles. Most difficulties in a given procedure can be avoided by repeated "dry runs" with detailed planning and organization.

When large animals are used, there are additional considerations as follows:

(*a*) The amount of radioactivity administered to the animal will be considerably larger than in the case of small animals.

(*b*) The large animals are difficult to restrain completely, and thus procedures must be such as to allow quantitative delivery of the activity by the chosen route in spite of movement by the animal.

(*c*) Contamination of the area by radioactive excretions must be avoided.

(*d*) Control should be such that under no conditions are edible animal products containing radioactivity used for human or animal consumption.

(*e*) Arrangements must be made for the disposal of large amounts of excreta and carcass material that may contain long-lived isotopes.

Preparation of the material for administration to the animal is carried out in accordance with the usual "hot"-laboratory techniques including use of barricades, localized vessel shielding, and remote-control devices. Delivery of activity to the animal presents the problem of avoiding total body exposure and particularly intense localized exposure to the hands of the personnel administering the radioactive material. Since remote-control apparatus has not generally been found practical for this application, there has been a tendency towards the use of the shielded syringe.

When pure beta emitters are used, simple syringe shields of Lucite are entirely satisfactory. It is determined, for instance, that the range of a 2-Mev beta particle in Lucite is only 8 mm (graph 5 in reference *17*). Pearl *et al.* (*18*) and Dudley *et al.* (*19*) have

described simple Lucite shields for this purpose. Figure 1 illustrates such a shield, for a 1-ml syringe, in use.

When gamma emitters are used, the shielding requirements are increased. Figure 2 shows a syringe shield which provides ¼-inch lead shielding and which has been satisfactory in operation at the multi-millicurie level. Designed by the Medical Division of the Oak Ridge Institute of Nuclear Studies, its outstanding features are the scale reflection and internal lighting which enable one to read the liquid level in the syringe or check for air bubbles without direct exposure to the radiation. The syringe can be handled without the hand's coming in contact with the shielding directly over the barrel of the syringe.

After the radioactivity has been administered to the animal, the problem of external radiation hazard is decreased. This is because the activity is generally distributed through a relatively large mass, and there is considerable absorption of the activity by the body of the animal.

With the metabolism units described in Part I, there is very little need for personnel to be near the animal once it has received the radioactivity. When the isotope is one that is excreted in large quantities, efforts should be made to handle the excretions with as little exposure to personnel as possible.

Perhaps the most commonly used dosing procedure involves the quantitative delivery of a radioactive solution either into a stomach tube or a vein. One method of accomplishing this involves the use of the apparatus shown in Fig. 3A. It is assembled from a metal 3-way stopcock and two syringes. In operation, the solution to be administered is measured into the open syringe 2, is drawn into the horizontal syringe 1, and, with manipulation of the stopcock 3, is delivered to the site of injection; wash solution is placed in the open syringe and the procedure is repeated. The advantages of this method are that small amounts of the active solution, delivered to the syringe by a micropipette, can be injected quantitatively, since it is not necessary to measure the volume by the syringe markings. Also, the rinsing of the system is conveniently accomplished without removing the needle from the site of injection, and the system can be well shielded if required, since it is never necessary to read the syringe markings.

Another method (*20*) which has been found convenient is illustrated in Fig. 3B. In this procedure, syringe 1, which is used for the wash solution, is connected to the injection needle or stomach tube by a convenient length of soft rubber tubing. In operation, the radioactive solution is injected into the lumen of the connecting tube with both dosing and wash syringes' being operated simultaneously. The dosing syringe 2 can be calibrated so that an accurately known volume of solution is administered.

In some instances, it may be desirable to deliver all of the radioactivity contained in the syringe rather than a predetermined amount. This can be done without removal of the syringe from the site of injection by rinsing it repeatedly with the wash solution supplied under slight positive pressure from the wash syringe.

Intravenous Administration

The most difficult procedure with farm animals is that of quantitative intravenous administration.

With small animals, which can be completely immobilized, the usual method of insertion of the hypodermic needle directly into the vein is satisfactory. This procedure is illustrated with the weanling pig in Fig. 1.

With larger animals, however, this method is made difficult because a slight movement of the animal may cause the

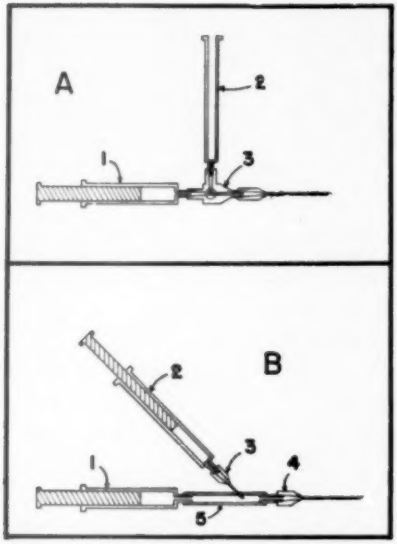

FIG. 3. Schematic diagrams of two methods for quantitative delivery of radioactive solutions

needle to go through the wall of the vein or be pulled out of the vein. There is no way of knowing just when this may occur; in such a case, an unknown portion of the radioactivity may be delivered in the tissues external to the vein. To avoid this possibility, the technique of venous catheterization can be employed. As discussed in a later section, there are also advantages in the use of catheterization when frequent blood samples from a given animal are required. The general procedure is the same whether the catheter is to be used for administration or blood withdrawal except that, in the latter case, the catheter is usually left in place over a longer period of time and some sort of protective covering is required.

Venous catheterization. This technique has been used with man and beast since 1929. Ralston *et al.* (*21*), Jarrett (*22*), and Rhode *et al.* (*23*) have reviewed the literature and described techniques suitable for intravenous administration of solutions and frequent withdrawal of blood from cattle, sheep, and dogs.

Various materials have been used for venous catheters (*21*, *24*) with preference being expressed for nylon, and polyethylene or polyvinyl plastics. Polyvinyl tubing has been used almost entirely in this laboratory. It produces minimal irritation to the tissues and has such desirable characteristics as heavy walls, translucency, resistance to animal heat, ease of sterilization, and nonwetting smooth surface.

With catheterization, the probability of thrombosis or embolism must be taken into consideration. Ralston *et al.* (*21*) reported that in studies using 19 catheters with 5 different cows, there was only one case of thrombi in the jugular veins and this was with an animal slaughtered 18 days after the insertion of the catheters. Anderson (*25*), using polyvinyl tubing in humans, reported that the average patient may be expected to develop thrombophlebitis as a result of about 3 days of continuous fluid therapy by this procedure. Cresson and Glenn (*26*) reported that polyethylene catheters caused a clotting reaction in dogs, but in only 1 out of 20 did a complete occlusion occur and this at 60 days following insertion. Some individual cases of thrombophlebitis have been observed in cattle and swine catheterized in this laboratory over periods of 10 to 30 days. If the catheter is allowed to remain indwelling for periods of several days, therefore, the possible development of this complication must be anticipated.

Catheterization is performed by threading a desired length of sterile catheter tubing through a hypodermic needle previously inserted into the vein. The needle is withdrawn and local pressure applied for hemostasis. A piece of sterile gauze beneath lengths of adhesive tape is used to anchor the tube in position. The catheter is immediately washed out with heparinized

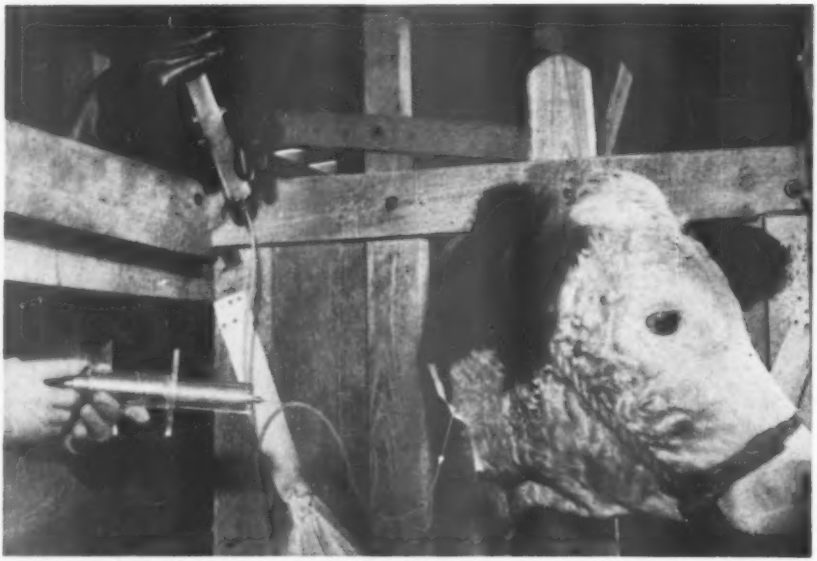

FIG. 4. A radioactive solution is being injected through a catheter previously inserted into the jugular vein

normal saline solution (4 mg heparin per 100 ml saline) from a syringe adapted with a blunt needle, and the tube tied off or plugged (*24*) until used.

If the catheter is to be left in place for any considerable period of time, a protective canvas or webbing strap cover is glued in position about the protruding end of the tube as a shield to make sure that the catheter is not pulled out by contact of the animal with the restraining stanchion.

Aseptic practices are followed throughout the operation, which include clipping, shaving, thorough cleansing of the area near the site of injection, and the use of sterile instruments to avoid possible infection. If needles larger than No. 15 are used in the operation, the animal is anesthetized locally with 1% procaine or other appropriate anesthesia.

Procedure. The actual procedure for intravenous administration is then a combination of the operations previously described in detail (Fig. 4). The animal is oriented in the metabolism unit and catheterized. The time between catheterization and administration of the radioactive solution should be minimal.

A large syringe containing heparinized saline or other wash solution is connected to a convenient length of soft rubber tubing to which a blunt hypodermic needle is attached with an adapter. This blunt needle is inserted directly into the open end of the catheter. The shielded dosing syringe containing the measured amount of radioactive material is then brought up and the solution injected into the lumen of the rubber tubing or directly into the lumen of the catheter itself. The dose is thus delivered quantitatively into the blood stream by the wash solution from the large syringe.

The needles are then removed and the catheter tied and left in position until the administered isotope has had an opportunity to become more or less uniformly distributed throughout the

circulation; this may vary from ten minutes to an hour after dosage. Immediately after injection, the blood in the direct vicinity of the catheter will have a high concentration. Therefore, if the catheter is removed immediately there will be the possibility of loss of the delivered dose and contamination by bleeding. The catheter and injection area can be monitored immediately after removal to ensure that no significant part of the dosage has escaped entrance into the blood stream.

Intraperitoneal Administration

When the radioisotope is to be administered intraperitoneally, essentially the same techniques and equipment already described can be used. A needle or catheter can be inserted into the abdominal region of the immobilized animal and the activity delivered quantitatively into the body cavity with normal saline or other wash solution as previously described.

The desired point of administration in cattle, sheep and swine is 6–8 inches anterior and ventral to the point of the right hip.

If it is impractical or impossible to immobilize the animal completely, a dosing catheter (0.055-in. I.D.) rather than a needle is the preferable administration method. The sterile catheter is threaded through a No. 13 needle into the body cavity and the needle removed leaving the catheter in position. A syringe with wash solution is attached to a length of rubber tubing which, in turn, is fitted with a No. 17 blunt needle that is inserted into the end of the catheter and the activity administered to the animal as previously described for intravenous procedure.

Oral Administration

In nutritional studies, it may sometimes be desirable to incorporate the radioactive material into the feed of the animal, to be given according to the usual feeding practices. When this is done, however, there is the difficulty of determining exactly how much of the feed the animal is getting. Also, there is always the possible hazard involved in the spreading of the radioactive feed around the unit and area. In some cases, however, where an animal has been trained to consume its feed quantitatively, this may be accomplished. It is usually more satisfactory to administer the radioactivity either as a solution or solid by capsule or directly by stomach tube.

In general, the capsule method offers an advantage over the stomach tube. The stomach tube requires thorough washing to insure quantitative delivery of the activity, and there is a greater possibility of spread of contamination. The capsule can be used only when the volume of the radioactive solution to be administered is compatible with the capsule size for the given animal.

It is necessary to prevent softening of the gelatin capsule between the time when the solution is placed therein, and the time when the capsule is administered. This may be done either by placing an inert absorbent such as filter paper in the capsule prior to addition of the solution or by using two capsules, the smaller containing the solution being placed within a larger capsule. These procedures will allow five to ten minutes working time.

The balling gun (see cover picture) is customarily used for oral administration; its use makes it convenient to place the capsule, ready to be loaded, in the gun and add the radioactivity by pipette. The cover can then be placed on the capsule with tongs and the gun handled directly with the gloved hand at a distance of 10–15 inches from the activity, which reduces exposure. In the case of beta emitters the walls of the balling gun serve as an effective shield. The mouth of the immobilized animal is opened, the balling gun inserted, the

FIG. 5. Oral administration of radioactive solution to a sheep by stomach tube, demonstrating the use of an inner polyvinyl tube to minimize surface contamination

capsule delivered in back of the tongue, the gun removed, and the animal's mouth held closed until the capsule is swallowed. A strong beta or gamma emitter can be followed with a monitor to indicate when the capsule has moved into the stomach.

The procedure is one that must be practiced by the individual to insure satisfactory results with the various species.

When a stomach tube is used, the diameter and length is determined by the size of the animal. Stiff-walled plastic or rubber tubing, or conventional animal or human stomach tubes may be used. With small volumes, it may be convenient to insert and clamp a small-diameter catheter inside the stomach tube to carry the activity and wash solution, thus decreasing surface area and adsorption.

The animal is restrained, the mouth spread, and the moistened tube inserted gently over the back of the tongue and into the esophagus. It should then be determined that the tube is actually in the stomach and not in the lungs. This can be done by placing the open end under water. If the tube is in the lungs, bubbles will be observed. Upon assurance that the tube is correctly located, a large syringe filled with wash solution is attached to the open end, the radioactive solution injected into the lumen of the stomach tube or insert, and the procedure carried through as previously described. Figure 5 illustrates the method.

Procuring Blood Samples

One advantage often quoted in the use of farm animals in experimentation is that relatively large quantities of blood may be taken frequently for analysis during the course of the study without producing an adverse effect. However, if the blood samples must be taken frequently, that is, every few minutes or perhaps once per hour over a day or so, it is difficult to avoid injury to the vein regardless of the skill with which conventional bleeding may be done.

Experience has indicated that frequent bleeding with a needle will upset the animal and may even interfere with normal metabolism. In such cases there is a definite advantage in the catheterization procedure. This is accomplished as previously described by

FIG. 6. Illustrating withdrawal of blood from the double catheter previously inserted into the jugular vein of a steer. Note protective canvas cover above syringe has been detached for access

insertion of the appropriate bleeding needle in the vein, threading a length of 0.055 or 0.085-inch catheter tubing through it, and removing the needle. The tubing is filled with heparinized saline and tied or plugged until a blood sample is desired.

The catheter should then be secured to the animal and covered with a protective shield to prevent its inadvertent removal by the movements of the animal.

A double catheter or "bypass" of the jugular vein has been used in cattle to facilitate routine periodic blood sampling and measurement of a strong beta or gamma emitter in the blood stream in vivo. See Fig. 6. The artificial collateral vessel was constructed from two 15-mm lengths of 0.085-inch polyvinyl tubing. One length was threaded anteriorly and the other posteriorly through a No. 13 bleeding needle and the two joined ends of the tubes connected by a short length of clear soft rubber tubing glued over the connecting ends. Another length of stiff clear plastic tubing was placed over all to protect and give rigidity to the connecting ends. A normal saline heparin solution was used initially to displace the blood in the tubes to prevent clotting. This bypass has functioned successfully for routine short-interval blood samplings for periods of 10–15 days.

When the number of blood samples to be taken over a short period of time is not too great, the decision on which method to use rests on the possibility of thrombus development due to catheterization as balanced against the effects of trauma produced by conventional bleeding techniques.

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Instrumentation for I^{131} Use in Medical Studies*

By Benedict Cassen,† Lawrence Curtis,† Clifton Reed‡ and Raymond Libby§

Scintillation counters have proved useful for the detection of gamma radiation from tissue. Several types of such counters have been designed at this laboratory to detect the gammas from I^{131}. The success of one of these for delineation of thyroid glands has led to the construction of a device for automatic scanning of the gland area and recording of the result

1. Localizing Counter

A scintillation counter specially designed in this laboratory for the localization of radioiodine in biological systems has been reported.¶ Further tests have shown that the intrinsic sesitivity of this counter is, in practice, greater than that reported.

Several improvements have been made, notable among them being the use of a better cement (Hyrax) for making optical contact between the calcium tungstate and the phototube envelope. With this counter it is easy to detect, with ¼-in. resolution, the boundary of a region containing only 0.2 μc of radioiodine per cm².

It has been shown possible‖ to delineate with fair accuracy the thyroid glands of patients given a dose of about 150 μc of radioiodine. (With this type of counter it is also possible to localize, readily and exactly, metastasized thyroid tissue in lymph nodes.) The procedure described here for delineating the thyroid glands is tedious but has been greatly improved by mounting the tube on a rack so that it can be moved in either of two directions at right angles to each other. This is done with lead screws turned by small hand cranks.

The outlines thus obtained were compared with the shape of the gland after removal by operation, or on autopsy, and were surprisingly accurate. The results indicated the desirability of an automatic scanning and recording device. Such a device has been built and is described in the last part of this paper.

2. Hand-Held Localizing Counter

A hand-held localizing counter that can be used for rapid determination of the boundaries of a thyroid gland, or other active tissue, by marking the outline on the patient's neck with a skin pencil, has been developed. This method is not as accurate as the point by point or scanning procedure, but is much less tedious and makes possible very rapid clinical location and classification of abnormalities and metastases.

* The information presented is based on work performed under Contract AT-04-1-GEN-12 between the Atomic Energy Commission and the University of California at Los Angeles. Section 5 of this paper has been reviewed in the Veterans Administration and published with approval of the Chief Medical Director. The statements and conclusions of the authors are the result of their own study and do not necessarily reflect the opinion or policy of the Veterans Administration.

Mr. Reed is co-author for Sections 1–4: Dr. Libby is co-author for Section 5.

† School of Medicine, University of California at Los Angeles.

‡ Formerly at the School of Medicine, University of California at Los Angeles. Now at the R. C. Scientific Instrument Co., Playa del Rey, California.

§ Radioisotope Laboratory, Wadsworth General Hospital, Veterans Administration Center, Los Angeles.

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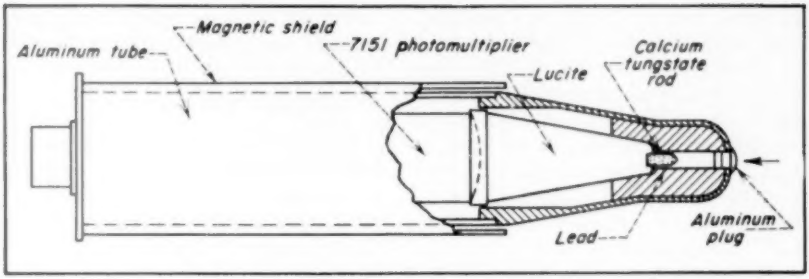

FIG. 1. Hand-held counter for tissue-boundary determination

Figure 1 shows this counter. An RCA 7151 photomultiplier tube is used. The scintillator is a single rod of clear calcium tungstate. The light produced is guided to the photosensitive surface by a conical Lucite light pipe that is cemented to the face of the phototube with Hyrax cement. This cement is also used to hold the calcium tungstate rod in place.

To obtain a sharp localizing effect with this arrangement, it is necessary to surround the calcium tungstate rod with a lead annulus of sufficient thickness and depth to shield most of the crystal from radiation coming in from outside a small angle. It is also necessary to surround the phototube with a mu-metal shield so the counting rate is not affected by the earth's or stray magnetic fields.

As a localizing detector, this counter is much more sensitive than the previous type. The over-all optical efficiency of the instrument is greater because the light can get to the photosensitive surface without reflections at air or vacuum interfaces. Also, the calcium tungstate rod is much closer to the layer of radioactive material. But the collimation of the gamma radiation is not as good and the spatial resolution suffers somewhat. If bias and amplification are set so that the background is about 4 cps, this counter, with a ¼-in. diameter circular aperture, is 3 to 4 times as sensitive as the earlier type with the same aperture.

Earlier, a modification of this type of detector was constructed using the larger 5819 photomultiplier tube and a much longer Lucite light pipe. The possibility was suggested of making radioactive the pontamine sky blue dye that Dr. Weinberg of the Long Beach Veterans Hospital injects into the mediastinal lymph plexus when making a radical removal of a lung. Usually the dye diffuses to the lymph nodes and makes those near enough to the surface visible so they can be excised in case of suspected lung cancer. Mixing radioactive di-iodofluorescein with the blue dye was suggested since with a gamma locater it would seem possible to locate more lymph nodes for excision, even if they were somewhat below the surface. Preliminary tests on rabbits have shown that the radioactivity of the di-iodofluorescein follows along with the visible dye.

For this application, the counter was made with a long light pipe so that the crystal and aperture could be introduced into a deep operation cavity. As yet it has not been used in a human operation.

3. Sensitive Wide-Angle Counter

Since a relatively small volume of calcium tungstate will absorb a high percentage of the incident 0.37-Mev

gammas from radioiodine, a scintillation counter can be built with an overall sensitivity from 30 to 100 times greater than that of an ordinary G-M tube. Specially designed G-M tubes can do better than the usually available types. However, it was thought worth while to investigate scintillation counters for use in thyroid-gland uptake and biological half-life studies with radioiodine.

By removing the lead shield from the counter discussed in Section 1 of this article, it was found that the thyroid-uptake studies could be made with an administered dosage of only 1–5 μc of radioiodine. The calcium tungstate crystals which were cemented to the face of the 1P21 tube were placed close to the patient's thyroid gland and a series of cases were followed.

Somewhat later calcium tungstate crystals were cemented to the face of a 5819 tube. The arrangement is shown in Fig. 2. Tests showed that 0.002 μc of radioiodine placed 1 in. from the face of the crystal assembly could easily be detected at about twice a background of 4 cps. Thyroid uptake and biological half-life studies could easily be made with 1 μc, or less, administered dose.

It appears likely that this tube is sensitive enough to make a direct measurement of radioiodine in tissue by placing the counter over a part of the body. In such use the crystals would have to be particularly well shielded from the thyroid gland. Measurements along these lines are in progress.

4. Counter Characteristics

In Fig. 3 performance characteristics of the wide-angle counter tube discussed in Section 3 are given. The data plotted are from a 0.62-μc I^{131} source at a distance of 40.5 cm from the face of the crystals.

It was found that, over a limited range of photomultiplier plate voltage, the net counts of a sample at a fixed distance from the counter are approximately the same for the same background count. To get the same background count when the plate voltage is raised, the bias must be raised. If the plate voltage is raised too much, the sensitivity to the sample will decrease somewhat, even though the bias is adjusted to give the same background.

The three sets of points in Fig. 3 are for three different fixed bias settings, the plate voltage being varied to obtain various background levels. Background counts is chosen for the abscissa because the sample count is relatively independent of the physical factors, as long as the background is set at the same value.

The net counts increase rapidly with low background and then flatten out. The ratio of gross to background counts reaches a maximum value and then slowly decreases.

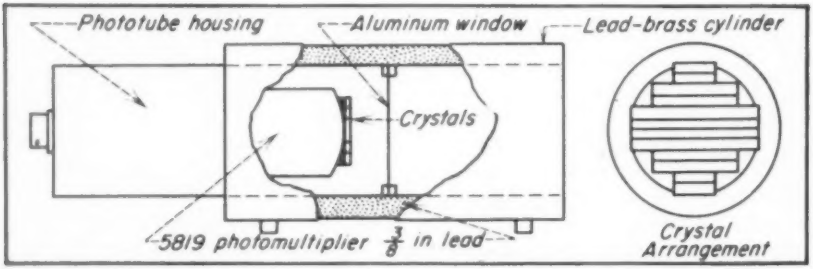

FIG. 2. Wide-angle scintillation counter designed for high sensitivity. The calcium tungstate crystals are cemented to the face of the photomultiplier

As is well known, the ratio of the standard deviation of the net counts, σ_{g-b}, to the net counts, $g - b$, is

$$\frac{\sigma_{g-b}}{g-b} = \frac{1}{\sqrt{g}} \frac{\sqrt{r^2 + r}}{r - 1}$$

where g = gross counts, b = background counts, and $r = g/b$.

The number of counts, C_p, required for a predetermined fractional accuracy, $p = \sigma_{g-b}/(g - b)$, is

$$C_p = \frac{1}{p^2} \frac{r^2 + r}{(r - 1)^2}$$

The time, t_p, required for C_p counts is

$$t_p = \frac{C_p}{g} = \frac{1}{gp^2} \frac{r^2 + r}{(r - 1)^2}$$

The third curve in Fig. 3 is $p^2 t_p$ plotted from this equation, with values of g taken from the experimental curve.

Counting with this particular counter is most accurate, then, if the background is about 8 cps, although the accuracy does not suffer too much over quite a range of background settings. In some applications a high gross-to-background ratio would be more useful than maximum accuracy. For this counter the maximum ratio is at about 6 cps, which is practically the same setting that gives maximum accuracy. The stability of the counting rate with respect to plate voltage fluctuations is at its best at higher backgrounds, say from 10 cps up.

5. *Automatic Scanner and Recorder*

The scintillation counter described in Section 1 of this article has been used for *in vivo* delineation of thyroid glands. The procedure requires taking individual readings over a rectangular network of positions and then estimating the gland outline by drawing a line through positions of a given reading or range of readings. With a little practice it is possible to get extraordinarily good agreement between the outlines so obtained and the actual outline of the glands as found either post operatively

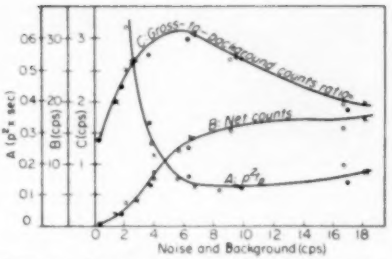

FIG. 3. Performance characteristics of a calcium tungstate scintillation counter

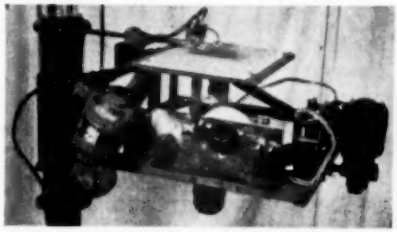

FIG. 4. Automatic scanner and recorder

or post mortemly. This procedure, however, is tedious and time consuming. An automatic scanner and recorder which would give a crude picture of the distribution of gland activity would obviously serve a very useful purpose.

The automatic scanner and recorder is shown in Fig. 4. The localizing scintillation counter is mounted on a carriage that is driven back and forth by a lead screw which in turn is driven by a reversing series motor operating through a reduction gear. The electrical reversing is accomplished by tripping a switch that is attached to the moving carriage, by a stationary but adjustable stop. The amplitude of back and forth motion can be adjusted by setting the positions of the stops. Each time reversal occurs the other motor is actuated for a small time interval. This interval is usually adjusted so that a scan is displaced about ⅛ in. from the previous one.

A small drawing board is rigidly mounted on the carriage that holds the counter. This drawing board is

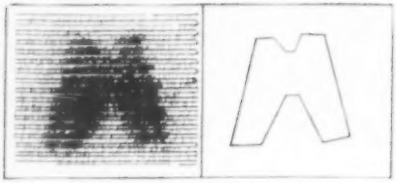

FIG. 5. Record of automatic scanning of filter paper soaked with 200 μc of I^{131} (left) and actual shape of the filter paper

scanned under an ink writing pen held rigidly on an arm attached to the main frame. The pen is attached to the arm of an electromagnet so that a short duration actuation of the magnet causes the pen to make a $\frac{1}{8}$-in. pip in a direction at right angles to the direction of motion of the scanning table. The pen and relay assembly was adapted from one used on Esterline-Angus time-marking recorders.

The amplified pulses from the photomultiplier tube are fed into a multivibrator regularizer and scaler. The scaler can be set to feed a pulse to the pen for any power of 2 up to 256 counts. Obviously, when the $\frac{1}{4}$-in. aperture of the detector moves over an active area the pips will be closer together. The higher the signal-to-noise count ratio the higher will be the visual contrast in the scanning record. If the contrast is sufficient the effect of a picture of the gland will be obtained. However, even when the contrast is low, a sensation of a picture can often be obtained.

The first tests of the scanner were made with filter paper wetted with a solution containing radioiodine. Figure 5 shows such a record and the corresponding outline of the filter paper which had been wetted with 200 μc of I^{131}. On this record, one pip corresponds to four counts.

The next tests were made on a frozen trachea preparation with attached thyroid glands obtained from a terminal patient given 3 mc of I^{131} 14 hours before death. The gland uptake in this time was approximately 15%. A series of records were obtained from 11 to 29 days after death.

The relative success of these tests made it desirable to extend the trials to *in vivo* mapping of thyroid glands in actual patients. Tests were made and typical results are shown in Fig. 6.

* * *

The parts of this program of investigation requiring testing on patients at the Radioisotope Units of the Sawtelle and Long Beach Veterans Hospitals were authorized by the Division of Medicine and Biology of the Atomic Energy Commission, and by the Veterans Administration. At the Sawtelle Hospital the clinical programs were undertaken by Dr. Herbert Allen, Jr. and Dr. Raymond Libby. At the Long Beach Hospital some other types of testing were done under the direction of Dr. M. Morton. Much of the clinical findings reflected back on improved instrumentation development, and important suggestions were made by the clinicians concerned.

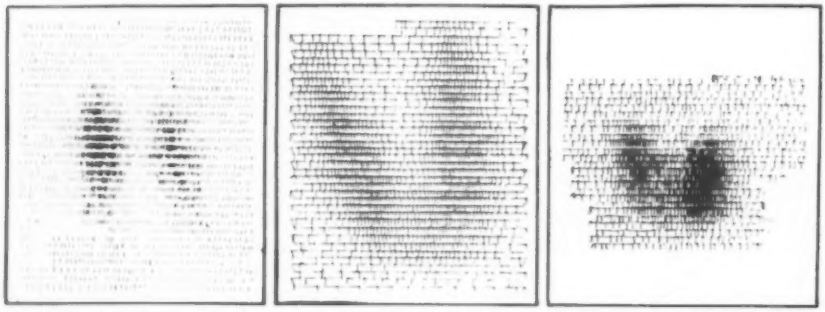

FIG. 6. Automatically recorded outlines of the thyroid glands of live patients

METHODS of FLUORINE and FLUORIDE ANALYSIS—III*

Beryllium compounds, aerosols, biologicals, glass, foods, and steel are just a few of the materials subjected to the methods of fluorine analysis noted in this series of articles for which the literature from 1816–1950 was surveyed

By F. E. McKENNA
Air Reduction Company, Research Laboratories†
Murray Hill, New Jersey

IN THIS LAST of three articles, the pertitanic acid and fluosilicic acid methods are discussed, the analysis of fluorocarbons, volatile fluorides, and elementary fluorine are considered, and spectrochemical methods, miscellaneous analytical methods, and the determination of fluorides in specific substances, such as foods, biologicals, and industrial materials are treated briefly.

Reactions of fluoride with peroxide-treated titanium solutions. An acidic solution of titanium after treatment with hydrogen peroxide exhibits a yellow color which has been attributed to the presence of pertitanic acid. This color is bleached by fluoride ions, but the bleaching is apparently not quantitativeiy proportional to the fluoride content (*300*).

The presence of alkali sulfates causes additional bleaching, while Al^{+++} and Fe^{+++} restore the color, but either a bleaching or a color restoration can occur in the presence of phosphates with a dependence on the relative concentrations of phosphate and fluoride. The optimum bleaching action due to fluoride occurs at *p*H 1.5, and there is a rapid decrease to zero effect at *p*H 2.5. The color-restoring properties of Al^{+++} and Fe^{+++} apparently indicate that the fluoride forms more stable complexes with them than with the titanium (*300–307*). The color-restoring action of aluminum is effected by the *p*H, but the restoration is not due to hydrolysis of the $Al(NO_3)_3$ solution.

At *p*H 0.9, sulfate and phosphate do not interfere (*308*). A 100-fold excess of nitrite does not effect the fluoride-titanium reaction (*205*). The color intensity of the pertitanic acid is decreased by lowering the temperature. When measured with a neutral wedge photometer (*309*), 1.00 ppm of fluoride in a 4% NaCl solution was analyzed as 1.03 ppm by its bleaching action.

Solutions of peroxidized titanium follow Beer's Law up to the solubility limits of the pertitanic acid, whether an excess of hydrogen peroxide is present or not, while Beer's Law is not obeyed in the fluoride-bleached pertitanate solutions (*310*). The bleaching action measured at 440 mμ is reproducible to 0.003% F^- and is accurate to 0.01% below 2.0% fluoride. The optical

* In Part I, published in the June NUCLEONICS (page 24) the lead chlorofluoride, calcium fluoride, aluminum, ferric, ceric, yttrium, lanthanum, and thorium fluoride methods of analysis were considered. In Part II, published in the July NUCLEONICS (page 40), discussion of the thorium fluoride method was continued, and analysis by the zirconium fluoride, zirconium lakes, and fluosilicic acid methods was considered. The isolation of fluoride as the volatile fluosilicic acid was also discussed.

† Certain portions of this paper were collected at the SAM Laboratories at Columbia University and at the Institute for Nuclear Studies of the University of Chicago.

density is an inverse linear function of fluoride concentration in the range of 0.6–3.2 γ/ml when $TiCl_3$ is oxidized by hydrogen peroxide (*312*).

Spectrochemical determination. The band spectra of the alkaline earth fluorides have been used for the estimation of the fluorine content of samples (*314*). The calcium fluoride band head at 5,291 A.U. can be used to detect 0.02 mg F^- (*315, 316*); less than 0.02 mg is difficultly recognizable. Visual comparison of the 5,291 A.U. band head for phosphate rock samples yielded a mean deviation of 16% from the actual content in the 0.05–1.7% F^- range, and a mean deviation of 12% by a log-sector comparison in the 0.3–3.6% range (*317*). The bands for calcium and strontium fluorides have detected as little as 2.5 γ of fluoride; the barium fluoride band is masked (*318*).

SiF_4 has been absorbed in a melt of 10 parts PbO and 90 parts B_2O_3 at 560° C; the silicon spectrum is then investigated in a portion of the melt. It was found that 40–50 γ of silicon corresponds to 110–120 γ of fluoride (*319–322*).

An electrodeless discharge through a gaseous sample in glass provides strong lines between 4,500–7,000 A.U. for fluorine compounds. The sensitivity limit is 0.001%, and the accuracy is 10% down to 0.01% fluoride (*323*).

Determination of gaseous fluorides and elementary fluorine. Fluorine in mixtures with HF has been determined by the weight gain of metallic silver or copper; small residues of unreacted gases are subsequently passed over silica to form SiF_4, which is determined as H_2SiF_6 by titration with KOH (*324*). Elementary fluorine has been determined volumetrically by reaction with liquid mercury, but this method is not suitable for micro amounts of fluorine (*325–329, 334*).

Small amounts of fluorine in the air can be measured by drawing the air through a solution of the zirconium-alizarin indicator, sulfuric and hydrochloric acids, for a colorimetric estimation (*206*). A zirconium-azoarsenate paper can be used to determine 2–40 ppm fluorides in the atmosphere (*330*). Fluorine reacts with heated NaBr to liberate gaseous bromine, which is determined colorimetrically at 4,250 and 5,250 A.U. (*332*). Small amounts of elementary fluorine in solution can be titrated with nitric acid with alizarin as the indicator (*333*).

Commercial hydrogen fluoride has been analyzed by the lead chlorofluoride method (*292*).

Determination as bismuth and antimony fluorides. Unlike other bismuth compounds, BiF_3 is not hydrolyzed when in contact with water; it is easily filtered and washed. Although BiF_3 is soluble in most acids, it can be precipitated quantitatively at *p*H 6.5. In the analysis of 0.2-gm samples of KF·HF, an average deviation of ±0.12% was obtained. A bismuth analysis of the presumed BiF_3 precipitate indicated 78.8% bismuth (78.5% calculated). For the most precise results, the precipitation should take place in a Bakelite or silver beaker. Because bismuth chloride, bromide, iodide, sulfate, and phosphate are soluble and hydrolyzed, their anions must be absent (*335, 336*).

Precipitation of fluorides as SbF_3 results in high values (*337*).

Determination as boron trifluoride. Fluorides have been determined volumetrically as BF_3 (*60, 208, 338, 339*); fluorides can be titrated with boric acid to the methyl orange endpoint because of BF_4^- formation (*79, 234, 320*). Fluoborates can be decomposed by boiling with a $CaCl_2$ solution to precipitate CaF_2 which is weighed (*341a* and *341b*).

The analyses of boron trifluoride and its derivatives have been summarized recently (*341c*).

Determination as triphenyl tin fluoride. Triphenyl tin fluoride precipitates quantitatively from a 60–70% alcohol solution at *p*H 7–9, when a 2% solution of triphenyl tin in alcohol is added. The precipitate must age overnight at 0° C before it is filtered and subsequently dried at 110° C. The separation is quantitative only if the procedure is followed closely (*342, 343*).

Determination with benzidine. Fluoride is precipitated when benzidine and mercury (II) succinimide are added; the precipitate is $Bz_2(HF)_4 \cdot HgF_2$ (*344, 345*).

Determination as a complex uranium fluoride. When an alkali fluoride is titrated with uranium (IV) sulfate, $UF_4 \cdot MF$ (where M is the alkali metal) is precipitated. The endpoint can be determined potentiometrically ($U^{+4} \rightarrow UO_2^{++}$) when the solution is buffered with sulfanilic acid and its potassium salt, i.e., *p*H 3.6. The method is reported to be sensitive to a few micrograms of fluoride (*78, 346, 347*).

Determination as a complex molybdenum fluoride. The bleaching action of fluorides on molybdenum compounds is the basis of a volumetric indicator that contains ammonium phosphomolybdate in an acetone solution (*348*). Silicomolybdic acid has been used colorimetrically to determine fluosilicates in fluorides (*349*).

Colorimetric determination as lead sulfide. Hydrogen fluoride and fluosilicic acid generated by the action of sulfuric acid on the sample in a gold container are absorbed in fused KOH. Barium fluoride and barium sulfate are then precipitated from a heated solution of the potash melt; after filtration, the precipitated barium compounds are treated with sulfuric acid, and the HF evolved is absorbed by lead glass. When the PbF_2 is completely dissolved in a saturated $KClO_4$ solution, gelatin is added and colloidal lead sulfide is precipitated and determined colorimetrically.

This method has been used for fluoride determinations in water, minerals, foods, and biological tissues (*350, 351*).

Colorimetric determination with methemoglobin. After a fluoride is added to methemoglobin, the optical density of the fluoromethemoglobin is determined between 6,100–6,200 A.U. (*352*).

Bleaching of dihydroxymaleic acid-titanium complex. It has been suggested that the bleaching action of fluoride on the titanium-dihydroxymaleic acid complex may be a quantitative method for fluoride (*353*).

Acidimetric-alkalimetric titrations. Commercial sodium or potassium hydrogen fluorides have been analyzed more satisfactorily by a titration with 0.1*N* NaOH in ceresin-waxed beakers with phenolphthalein as the indicator than by a calcium fluoride precipitation. Apparently no H_2SiF_6 was formed because the endpoint did not fade (*354*).

Determination by the enzymatic inhibition due to fluorides. Fluorides, particularly in foods, can be determined by the decreased activity of lipase as a hydrolytic agent in their presence (*355*); potato phosphatase can also be used (*356*). Respiration and fermentation are more sensitive to fluorides in starved yeast cells than in normal cells; the fluoride inhibition is temperature

dependent and is also affected by the presence of air (*357*).

Wettability of glass. Measurement of the contact angle for a CrO_3 solution containing fluoride is reproducible to ± 1° between 30° for 0.003 mg NaF per liter to 82° for 1.5 gm per liter. Only boric and silicic acids interfere (*257*, *358*).

Determination of fluorine in organic fluoro-compounds. Combustion of organic fluorides in a bomb with 25 atmospheres of oxygen does not yield correct values if either moisture or alkalies are present; if the combustion occurs in the presence of KI and KIO_3, followed by titration of the liberated iodine with thiosulfate, an accurate estimation of the fluorine is possible (*359*).

Volatile organic fluorides have been passed over silica or silicic acid at 900° C; the SiF_4 thus formed is absorbed in an aqueous solution of sodium hydroxide (*360*). Any $SiOF_2$ that may be formed has been decomposed by a subsequent flushing with hydrogen (*361*).

Halocarbons which do not contain hydrogen are decomposed in the presence of quartz powder at 1,000° C in a stream of oxygen; the fluorine reacts with quartz to liberate SiF_4 which is adsorbed on alumina at 175° C. If chlorine or bromine are liberated simultaneously, they are absorbed by metallic silver at 295° C and then weighed as the silver halides (*362*). After burning fluorine-containing organic compounds in oxygen between 450° and 900° C in the presence of quartz, the SiF_4 produced has been absorbed in a KF solution (*363*). Volatilized fluorocarbons are passed through a platinum tube at 1,100° C with nitrogen and moist oxygen. The HF formed is collected in water and determined colorimetrically with peroxidized titanium (*364*).

Fluorinated liquids or solids containing carbon, hydrogen, oxygen, and fluorine can be burned at 1,250° C in an atmosphere of oxygen and water vapor in a platinum tube. The HF is absorbed in water and determined alkalimetrically (*365*). Compounds containing both fluorine and sulfur can be burned with hydrogen in a platinum boat supported in a plastic apparatus; the HF is determined after absorption in a dilute KOH solution (*366*).

Fusion with oxidizing mixtures, such as sodium peroxide, potassium chlorate and sugar, or sodium peroxide alone, has been used to cause decomposition of the organic compound to yield inorganic fluorides. Errors of ± 1% have been found in some of these methods (*67*, *367–370*).

Certain organic fluorine compounds have been reported to be decomposed at 500–550° C with sodium carbonate (*371*); fluorine in aliphatic compounds or in the side chains of aromatics can be converted to inorganic fluoride by heating with CaO or $CaCO_3$ in a nickel tube for two hours at a yellow heat. Fluorine attached to an aromatic nucleus requires a temperature of 1,000° C for decomposition (*6*, *322*).

When samples have been heated in Pyrex with potassium nitrate and concentrated sulfuric acid, both SiF_4 and H_2SiF_6 escape. The weight loss is considered to represent the fluorine in the compound (*373*).

Nonvolatile fluoro-compounds have been decomposed with sodium in liquid ammonia, ether, or tributylamine (*360*, *367*, *374*). Sodium and potassium fusions at 400° C have been used, but the fluorine has been found completely only if the fusion occurs in an evacuated tube (*375*, *376*). A potassium fusion (*377*) in a nickel bomb at 500–550° C for two hours, followed by a distillation of SiF_4 and a thorium titration, have produced less than 0.5% deviation from the theoretical.

Chlorine was determined simultaneously by the Volhard method, or by titration with aluminum chloride with Eriochrome cyanine R as the indicator (*378*).

Organosilicon fluorides do not react with sodium in liquid ammonia; decomposition can be affected by a sodium peroxide fusion (*379*).

Hydrogen fluoride has been generated by vaporization of the organic compound in a stream of hydrogen and subsequent combustion of the gas stream (*380*) or by the lamp method (*381*) followed by a thorium titration or a lanthanum precipitation.

Traces of fluorine in organic compounds have been determined by vaporizing the sample over platinum at 600–800° C (*382*); the inorganic fluoride is then determined by a thorium titration.

Determination of fluorine or fluorides in specific materials. Some of the methods described above have been adapted to the analysis of specific materials: aerosols (*383*); baking powders (*384–386*); beryllium compounds (*387, 388*); biologicals (*241*); blood serum (*389*); bones and teeth (*312, 390–395*); borates (*152, 396*); cryolith (*397, 398*); enamel (*399, 400*); feedstuff (*401–403*); fluorspar (*404–409*); foods (*402, 410–419a* and *b*); gelatin (*420*); glass (*311*); human tissues (*119, 123*); insecticides (*41, 296, 421–423*); lime (*276*); meat (*424–426*); minerals (*92, 390–393, 395, 427, 428*); phosphates (*90, 429–438*); plants and animals (*16, 120, 242, 356, 394, 439–442*); chromium plating baths (*443*); table salt (*444*); sardines (*445*); silicates (*431, 446*); slag (*447*); soils (*103, 391, 448–454*); steels (*455*); tannery solutions (*456*); toxicological methods (*457*); waters (*92, 104, 155, 215, 395, 403, 458–473*); wine or beer (*46, 356, 394, 427, 474*), wood (*475–477*); wood preservatives (*478*); wool (*479*); zirconium (*480*); miscellaneous (*219, 245, 258, 259, 481–513*).

When biological materials are to be ashed before a fluoride determination, there is danger of fluorine loss during the ashing process. MgO_2 has been used with fair success, and excellent results have been obtained with magnesium acetate prepared from electrolytic magnesium (*181, 241–244, 514, 515*). If CaO is used, it should be remembered that the CaO may contain 150–200 mg fluoride per 1,000 gm; such fluoride can be removed by a distillation from perchloric acid (*516*). Aluminum nitrate has been used successfully at 600–650° C as an ashing agent even with organic acids present (*313*).

Summation

No universal scheme can be set forth for the analysis of fluorine-containing materials. The amount of fluoride as well as the matrix must determine the analytical methods to be considered. In all cases the proficiency of the analyst with the method or technique adopted is of major importance. Certain contradictions (which are apparent in this collation) as well as personal experience have indicated that all analysts are not equally adaptable to all of the techniques suggested in the literature.

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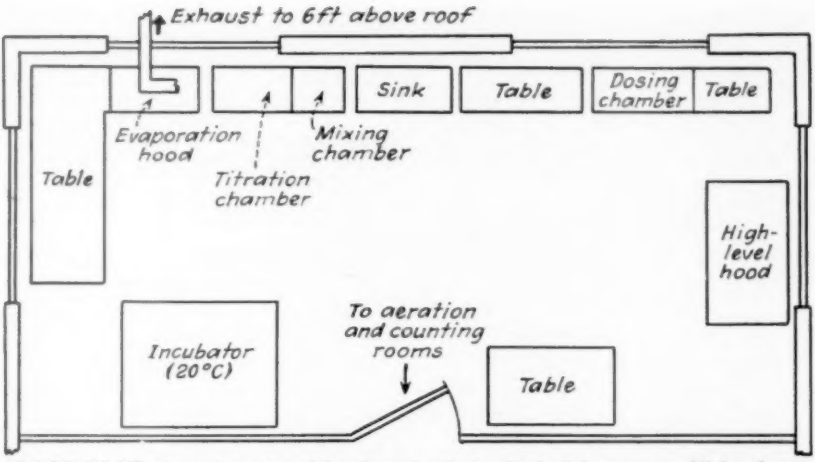
"Sea disposal of solid wastes is an important and satisfactory method. However, a number of factors must be taken into consideration. These include international clearances, avoidance of shipping lanes, oceanography, including currents, depth, animal and plant life, and proximity to land masses. The solubility and dispersibility of the wastes must also be considered. Preference is naturally for encasement of the material in concrete and disposal in canyons or deeps of the sea."

—C. C. Ruchhoft, in lecture at Inservice Training Course in Radiological Health, Ann Arbor, Mich., Feb. 7, 1951



FLOOR PLAN shows location of hoods and other units in laboratory. All hoods are connected to exhaust ducts. High-level hood is modified chemical hood; incubator is a walk-in type of refrigerator maintained at 20° C

Redesign of a Sanitary Engineering Laboratory To Permit the Use of Radioisotopes

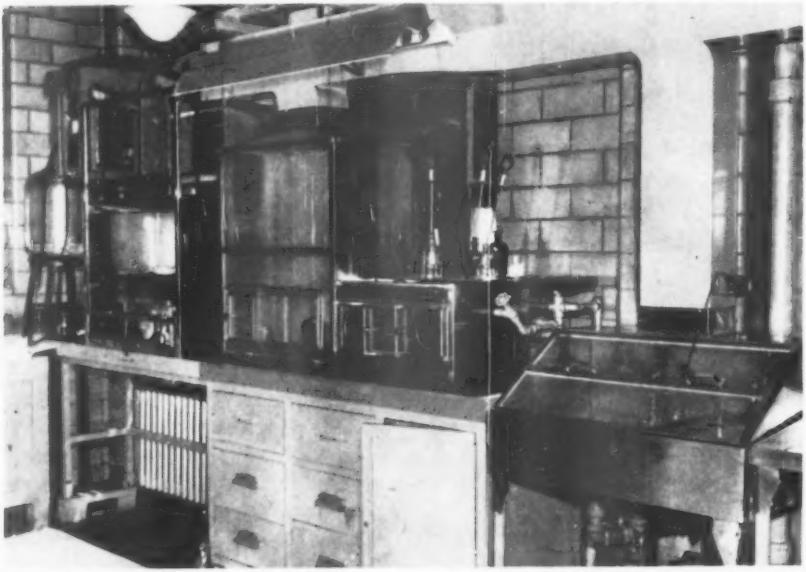
Biochemical oxidation studies employing radioisotopes can be safely and successfully carried out at the millicurie level if work is done in hoods to minimize contamination. An NYU laboratory has been equipped specifically for these studies

By WERNER N. GRUNE* and PAUL B. KLEVIN†

THE FACILITIES of the Lewis Van Carpenter Research Laboratory of New York University have been used for diversified sanitary engineering research involving sanitary chemistry, bacteriology, and biology. But the existing laboratory facilities were inadequate to insure safe handling of radioisotopes during an AEC-sponsored study of the course of biochemical oxidation of sewage containing P^{32} and I^{131}. Since relatively large quantities of these radioisotopes would be handled in solution and since high accuracy of determination had to be maintained in radiochemical analytical procedures, it was of paramount importance that the laboratory be redesigned so that experimental procedures could be carried out in an enclosed and ventilated

* Waste Disposal Research Project, Sanitary Engineering Laboratory, New York University, New York, N. Y.
† Health and Safety Division, New York Operations Office, U. S. Atomic Energy Commission, New York, N. Y.

BEFORE Porous work surfaces, unshielded and uncompartmented work areas, and open drainage troughs at back of benches made contamination easy, decontamination difficult in original laboratory. The laboratory was unventilated, and the personnel hazard was potentially very high because of the danger of inhalation of radioactive aerosols dispersed during shaking of BOD bottles and during other operations in BOD determinations



AFTER Enclosed stainless-steel hoods confine contamination, and their plastic fronts shield workers from beta radiation. Each step in BOD determinations is carried out in special hood which can be flushed with built-in spray. Ventilation ducts provide for correct air flow through hoods. Shown here, from right to left, are enclosed sink, mixing chamber, titration chamber, and evaporation hood. Size of hoods can be estimated from dimensions of titration chamber: 30 in. wide, 15 in. deep, and 38 in. high. Construction and use are described in text

BOD Test Procedure

The biochemical oxygen demand of sewage is determined under radioactive conditions in the following manner:

1. Distilled water is aerated to increase the dissolved oxygen content and made up with iron and calcium chlorides, magnesium sulfate, and a phosphate buffer to simulate natural stream conditions.

2. Half of the prepared distilled water is drawn off and split into quarters.

3. One quarter is dosed with nonradioactive reagent for a control. The others are dosed with 0.1, 1.0, and 10.0 mc/l respectively.

4. Cotton-filtered domestic sewage is added to the remaining half of the distilled water to the extent of 4% by volume, and a set of quarters is made up.

5. The sewage-dilution quarters thus prepared are dosed as in step 3.

6. Duplicate 275-cm^3 samples are taken from each quarter each day for 20 days. BOD test bottles are used for the samples.

7. After suitable incubation in the refrigerator, the samples are analyzed according to a modified Winkler determination for dissolved oxygen (4). This involves adding $MnSO_4$, alkaline KI, and H_2SO_4, releasing iodine. A titration for the liberated iodine with $Na_2S_2O_3$ gives a direct measure of the dissolved oxygen.

system, thus minimizing contamination.

The scope of the study embraces the effects of radioactive substances on existing types of sewage treatment processes, on sewage systems, and on self-purification of streams and watercourses. Also considered is the broader aspect of mechanisms to remove isotopes from liquid wastes that are to be discharged into sewage systems.

The body of the experimental work consists of thousands of Biochemical Oxygen Demand (BOD) determinations. The BOD test procedure, detailed on this page, and incorporation of safe handling of the radioisotopes were the determining factors in redesign of the laboratory. However, cost was kept at a minimum.

The basic equipment for the radioactivity laboratory consists of work benches with specially designed glovebox hoods, a large laboratory exhaust hood for high-intensity handling of the radioisotopes, a walk-in type refrigerator equipped to handle large quantities of radioactive and nonradioactive BOD bottles, and a local ventilation system to remove contaminated air to 6 ft above the roof level. Existing designs of others confronted with similar problems served as guides and were adapted whenever feasible. Various publications provided information on hazards and safe handling of isotopes (*1–3*).

Hoods

All of the hoods were designed for a minimum air velocity of 100 ft/min into the maximum face opening. In addition, the hoods are equipped with drains for waste solutions, and the bottoms of the hoods are slightly pitched towards these drains. This allows decontamination and flushing of contaminants within the hoods.

A closed waste drainage system is provided, in contrast to the earlier open trough. With the closed system, evaporation with subsequent air contamination does not exist. This is shown by air monitoring for radioactive aerosols. Constant flushing of all hoods and other drainage dilutes the activity of the laboratory wastes to the background level of the survey meter (0.2 mr/hr).

A number of different hood types are used. Stainless steel of 18 gage was used for most construction.

High-level hood. A conventional chemical laboratory hood previously

Cost of Radioactivity Equipment

To adapt the laboratory to work with radioisotopes, equipment costing $1,900 was installed and instruments worth $1,800 were borrowed. The breakdown:

Four stainless-steel hoods, a completely enclosed laboratory sink and drain table (including all necessary appurtenances and plumbing)	$1,200
Ventilation for all hoods (including duct work and two blowers)	250
Modification of existing hood for high-level work (including ventilation, shielding, and storage cabinet)	450
Total laboratory equipment cost	$1,900
Instruments—two G-M type scalers, two portable beta-gamma survey meters and a portable air sampler were obtained on loan from the New York Operations Office of the AEC, would cost	$1,800

in use was improved to serve as a high-level hood for opening isotope shipments and preparing initial dilutions. A 6-in. blower of 585 ft^3/min capacity was installed to provide a minimum air flow of 100 ft/min across the 5.5-ft^2 face opening. The exhaust duct terminates 6 ft above the roof of the building.

All seams inside the hood were caulked to insure water tightness and to reduce contamination of the floor from spillage. Across the front of the bench area of the hood, a 1½-in. aluminum angle was installed to serve as a stop lip to prevent liquids from running over the edge. A water outlet and drain are provided within the hood for initial rinsing and cleaning of dilution pipettes and are also used for periodic flushing of the working surface.

To minimize air turbulence in the hood during experimentation, a sheet-metal canopy and baffle combination were added to create double walls and roof with approximately a 2-in. clearance through which the air is exhausted. The under side of the hood is completely enclosed with a wooden framework and lined with 6-mm glass for a beta shield. A storage vault of interlocking lead bricks installed within the glass cabinet serves both as a gamma shield and as a storage place for shipping containers, lightly contaminated gloves, glassware and other equipment until the activity has sufficiently decayed to allow safe handling.

For handling P^{32} the sides and face of the hood proper are shielded with transparent plastic sheets. This type of shielding combines excellent visibility with adequate radiation protection. For handling I^{131} the sides and face opening are built up with double $1 \times 4 \times 8$ in. lead brick. A mirror hung at an angle inside the hood provides visibility.

Dosing chamber. A second hood was installed for filling BOD bottles; this hood is shown in Fig. 1. The face of ½-in. Plexiglas is framed in stainless steel so that it can be raised and lowered. This is necessary only for periodic inspection, monitoring, and decontamination. Two 5-in. portholes through which rubber gauntlets are flange-fitted prevent contact contamination during the bottle filling. The hood absorbs all betas from P^{32} and I^{131}; gamma radiation from I^{131} through the hood measures less than 1 mr/hr.

A trap door on the right side of this hood is used for inserting and removing trays of BOD bottles. To prevent external contamination, from spillage during removal of the bottles and trays

FIG. 1. Dosing chamber for filling sample bottles. From 250-300 bottles are processed each week

FIG. 2. Lead-shielded separatory funnel used for transfer of radioisotope to quarters of sewage dilution under test

from the hood, a drainboard with 4-in. lipped edges is provided. Another safeguard is a shower spray located in the top of the hood so that filled bottles can be washed before removal from the hood.

Rubber tubing is inserted through the top of the hood for siphoning from outside carboys into the BOD bottles. The isotope is added to the sewage substrate in the carboys by using the lead-shielded separatory funnel shown in Fig. 2. The funnel is filled with the required isotope from a shielded Erlenmeyer flask into which the radioactive material is initially transferred within the high-activity hood. After filling, the separatory funnel is raised by using the chain and pulley system that supports it. Its contents are then transferred into the carboys.

Sink. The third hood is a stainless-steel double sink covered with ¼-in. Plexiglas in a stainless-steel frame. The split cover is piano-hinged in the middle so that when it is raised any radioactive drippings from the inside fall back into the sink. Foot-pedal control valves for the water supply prevent hand exposure to radioactivity.

This sink and the hoods described in the following paragraphs are shown in the "after" picture of the laboratory on page 60.

Mixing and titration chambers. These two hoods were constructed as a single unit with a sliding door between them. They are used to carry out the Winkler (*4*) determination for dissolved oxygen, under radioactive conditions.

The smaller of these two units is designed specifically for manual shaking of BOD bottles. For the purpose of Winklerizing incubated sample bottles, many types of mechanical and automatic mixing or shaking devices were investigated. None of these was found to duplicate the natural shaking action of the wrist, so it was necessary to install gauntlets in this hood. In this fashion, no spillage can find its way out of the hood during the vigorous wrist-shaking operation. The inside of the unit is kept free of contamination by use of the shower spray installed in the top of the hood.

The taller of the attached hoods is used for the final operation of titrating the liberated iodine as a measure of dissolved oxygen. After the BOD bottles are fixed in the mixing unit, they are moved directly into this hood

through the sliding separating wall. Two slide-covered openings on the face of the taller titration unit permit operation of the burette and other manipulations. Surgical gloves are worn during this work; gauntlets were unsuitable for handling precision equipment.

Evaporation hood. The sixth hood is used for evaporating radioactive water and sewage samples before analysis for ammonia, nitrite and nitrate. This hood is substantially the same as the others.

Working Surfaces

To prevent excessive absorption of radioactive substances by the surfaces of the laboratory furniture, a strippable Tygon paint was applied. This paint satisfies the requirements of low permeability, ease of application (paint brush), ease of removal, and economy. The same protective coating was applied to the interior of the refrigerator.

In addition to the strippable paint, a white absorbent diaper paper with a kraft backing is used to cover all working surfaces. This paper effectively absorbs accidental spills and prevents penetration to the surface.

After each experimental laboratory run, the absorbent paper is gathered up and discarded with other combustibles in the waste-disposal area. The laboratory benches are then checked by instrument, and any area that is found to be contaminated is stripped of the coating. A new coating is then applied.

All laboratory working surfaces are provided with 1½-in. aluminum angle edging to prevent dripping.

Handling Apparatus

To minimize hand and body exposure, all isotope manipulations are done with gloves and long tongs. A neoprene surgical glove is employed for general-purpose laboratory use and whenever gauntlets are not incorporated in the hood. Where gauntlets are installed, a 16 or 18 in. size of flexible, lightweight style, with special grippers to prevent slippage of glassware, are used.

Several types and lengths of stainless-steel tongs are used. Most suitable for general use are tongs that are 6, 12, or 18 in. long, but 30-in. tongs are needed for handling isotopes in the high-intensity hood. To prevent glassware slipping, tongs are rubber tipped.

Pipetting of radioactive solutions is carried out by remote handling. Two 30-ml biological syringes mounted on the exterior of the high-level hood are used to meter the dose.

Disposal of Wastes

As waste materials accumulate from the various processes and manipulations, they are separated into combustibles and noncombustibles. Separate step-on cans, properly identified, are provided for each type of waste. These cans are lined with waxed-paper bags which are transferred to a waste disposal area maintained in the basement of the building. There, they are stored in 30-gal fiber containers, clearly marked as to type of waste, radiation intensity, and specific isotope. All wastes are shipped to the AEC for ultimate disposal.

* * *

The authors wish to extend grateful acknowledgement to M. Eisenbud, W. B. Harris, and H. Blatz of the Health and Safety Division, New York Operations Office, U. S. AEC, for their constructive criticisms and helpful discussions.

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Development of Nuclear Reactors

In a talk before the Institute of Radio Engineers this spring, Dr. Alvin M. Weinberg, Director of Research, Oak Ridge National Laboratory, analyzed the pros and cons of high- vs low-temperature reactors and concluded that low-temperature power plants will be dominant. Here are excerpts from this talk

A NEUTRON CHAIN REACTOR is unique among sources of thermal energy in that the rate of energy release in a reactor is independent of the temperature at which the energy is generated. This basic independence of nuclear-energy release from temperature has allowed the development of reactors to proceed along two parallel threads which are characterized by temperature and purpose.

There is a low-temperature thread that is exemplified by the large-scale plutonium producers at Hanford, and there is a high-temperature thread exemplified by the projected naval or aircraft reactors. For a given total energy release, the required heat-transfer surface is smaller the greater the temperature drop. This, in substance, means that a compact reactor which produces useful power must be a high temperature one—and, quite apart from questions of thermal efficiency, this is the reason why the military mobile reactors belong to the high temperature genus.

On the other hand, where reactor size is unimportant, there is incentive to run at low temperature—both because at low temperature, materials are generally more adequate, and because the very best moderator, heavy water, is inherently a low-temperature material. In a conventional steam power station where the prime motivation is fuel economy, advantages in material performance which accrue from low pressure and temperature are more than balanced by the thermodynamic or fuel efficiency advantage of high temperature. In a nuclear power plant where fuel efficiency is secondary to plant efficiency the incentive to run at high temperature is much smaller.

In fact, it is my own belief that the dominant kind of nuclear power plant will be a rather low-temperature, low-thermal-efficiency system which will combine the long term objectives of fissionable material and power production, and in which all compromises will be made in the interest of over-all low cost. The fissionable material produced from such low-temperature power reactors will presumably be available for weapons or for high-temperature, high-efficiency mobile systems; but since the uses for the highly compact high-temperature systems will be specialized, it is my guess that in the foreseeable future it will be these rather low-temperature fissionable material power producers which will represent the main stream of nuclear energy development.

Such combination (Pu or U^{233} plus power) reactors would supply increasingly large amounts of the country's power. It is my belief that the development of networks of such dual-purpose plants is the main job of nuclear engineering, since it is by successful operation of such networks—more than by the successful propulsion of military vehicles—that the long term importance

of nuclear energy can be demonstrated.

There is the rather practical question—in deciding on a power producer—of whether it should be a breeder or a converter—i.e., should it convert newly dug U^{235} into Pu^{239} or U^{233}, or should it increase the total supply of U^{233} or Pu^{239}? That breeding is the ultimate choice is clear, since eventually we will run out of U^{235}. But the cost of breeding, per atom of newly bred material, should be higher than the cost of conversion, and therefore a balance must be struck between the cost of digging less enriched ores against the cost of producing expensive bred material. The question of whether it pays to invest one's money in a breeder or an additional ore-processing plant is by no means easy to answer. At some price for a pound of uranium ore it makes more sense to breed rather than to convert. What that price is I don't know.

Granted that some kind of nuclear reactors which will produce power and fissionable material are of long term significance—what about the other large scale engineering systems—the isotope separators—will they occupy any long-term important position in nuclear energy development?

While it is too early to say, I think there is a fair chance that isotope separators will be important even in the long run for the following reason: a chain reactor using natural uranium stops when a rather small amount of its U^{235} is burned up. The problem then is whether it pays to dig new uranium out of the ground or whether it pays to ship the depleted uranium back to the diffusion plant to reenrich it. Just as in the choice of breeders vs converters, so here cost of the ore determines whether it pays to reenrich. Since, as high-grade ores are worked out, raw U will almost certainly increase in price, it is possible that reenriching will prove of long term importance—i.e., isotope separation will possibly retain its significance.

Thus a nuclear power plant system might include an ore plant which supplies normally enriched U to a large central power plant which, after burning part of the U^{235}, returns its waste product to a diffusion plant for reenriching. The economic engineering of such a complex will clearly depend on an integrated analysis of the whole reactor-diffusion plant complex—which takes account, say, of the fact that part of the power from the reactor must be fed back into the diffusion plant.

"It was frequently hinted in the early days that the pile made obsolete the use of the cyclotron in the production of artificial radioactive isotopes, but this fear has proved to be misleading and the two instruments are complementary. The pile cannot make the extensive variety of radioisotopes producible with a cyclotron because the latter makes use of a much greater diversity in energy and type of nuclear bombarding particles. The pile is a copious source of medium energy and slow neutrons, the latter giving rise to large yields of isotopes only by (n,f) and (n,γ) reactions. The cyclotron produces many isotopes having a deficiency of neutrons. The pile tends to add neutrons."

—W. V. Mayneord in "Reports on Progress in Physics," Vol. XIV (The Physical Society, London, 1951)

BOOKS

Radioactive Tracers in Biology (2d edition), by Martin D. Kamen, Academic Press Inc., New York, 1951, xiv + 429 pages, $7.50.

Carrying the subtitle "An Introduction to Tracer Methodology," this 429-page volume replaces a first edition comprising 281 pages published a little over three years earlier. Besides the extra 150 pages, the second edition contains more tabular material and is, in general, more concisely written.

A comparison of these two editions, serves to characterize the rapid development of interest by biologists in the field of radioactive tracers and indicates further that the three-year interval between editions was productive in publications and data pertinent to radiobiology.

The second edition continues the excellent plan of dealing with the more important individual isotopes in separate chapters.

The addition of five sections of an appendix will be found most useful and should be well received.

A brief discussion on radiation hazards and dosage calculations which is included is helpful, but it might profitably be expanded.

The problem of placing between the covers of one book significant information on radioactive tracers in biology can be solved only by a compromise between limitations of space and selection of useful material. For many workers, this volume will provide the essential introductory information. It is clearly written, for the most part, and represents an earnest attempt to provide for biologists a useful basic text.

—JAMES H. JENSEN

Head, Plant Pathology Department
North Carolina State College

Opportunities in Atomic Energy, by Karl D. Hartzell, (Vocational Guidance Manual) Grosset & Dunlap, New York, 1951, 144 pages, $1.

In April, 1950, the Atomic Energy Commission figured that job opportunities in the atomic-energy program in the United States would break down as follows:

Scientific and technical	12%
Management and supervisory	7%
Administrative	20%
Skilled	26%
Semiskilled	18%
Unskilled	17%

The story of the kind of work to be done and found in these categories is a very interesting one, and it is expertly told in this well-documented vocational guidance manual. The author, who is in charge of Educational Services for Associated Universities, Inc., contractor for Brookhaven National Laboratory, is to be complimented on his up-to-date statistics and sensible evaluation of future trends in the field.

"What atomic energy is" is explained early in the book. Then the American program for atomic-energy development is discussed in all its phases—administrative (AEC), technical, and military.

Thirteen pages are devoted to personnel problems and policies of the AEC and its contractors. This is followed by 32 pages about the placement possibilities for scientists and engineers in the program, 12 pages for managerial, supervisory, and administrative personnel, and 5 pages for skilled, semiskilled, and unskilled workers.

In a very enlightening chapter on personal, educational, and experience requirements, the author dispenses a variety of information on, for instance, salaries, fellowships available, employment application procedures, etc.

A comparison of salaries for pro-

fessional scientific employees paid by AEC labs with those paid by industrial labs shows a smaller difference than that shown by a comparison of salaries within seven different AEC labs. The author, in connection with the latter statement, found that "the weighted averages of both B.S. and Ph.D. monthly salaries, by years since the B.S. over a 20-year period, place Los Alamos at the top with $457. The others follow in order: Westinghouse $451, Argonne $423, Sandia $414, Brookhaven $405, Hanford $351, Ames $290."

This manual is more than worth its price to anyone considering a career in the atomic-energy field. —*E.P.*

Industrial Medicine on the Plutonium Project—Survey and Collected Papers (Div. IV, Vol. 20, of National Nuclear Energy Series), edited by Robert S. Stone, M.D., McGraw-Hill Book Co., Inc., New York, 1951, xxiv + 511 pages, $6.25.

This is a remarkable book on the medical program for industries of the Plutonium Project.

To evaluate properly the enormous task of caring for the workers, as well as searching for methods to overcome serious hazards, one should first read the second-last chapter of the book, *Status of Health and Protection at the Hanford Engineer Works*. This document, the editor explains, "was written for the employees of the Hanford Engineer Works shortly after the dropping of the atomic bombs on Japan. Many of the workers at the plant had been unaware of the nature of the materials with which they were working; this document was prepared to reassure them concerning their safety on the job and to emphasize the fact that great care had been taken to safeguard their welfare." One may well imagine how amazed these workers were when they suddenly learned of the dramatic developments they had unknowingly contributed to. One cannot help having a similar feeling of amazement when reading this book.

The book is divided into two parts, "Survey" and "Collected Papers." Each part contains ten papers, written by Dr. Stone, the editor, and eighteen well-known contributing authors. The first part describes the development of medical services for a vast project handling hitherto unknown large quantities of radioactive substances. Methods for protecting the personnel against various hazards were worked out and reliable values for maximum permissible exposures, both external and internal, to all kinds of radiations were ascertained. Detailed studies are included on the effect of ionizing radiations on blood, on distribution and excretion of uranium and plutonium, and on management and treatment of persons exposed to radiation. Requirements of an adequate health service in relation to atomic research and industrial development are enumerated.

The second part of the book contains a number of important studies regarding radiation effects. Of special interest are those on the effects of total-body exposures to X-rays, and tolerance to whole-body irradiation of advanced-cancer patients. Treatment of plutonium poisoning by metal displacement is an equally important contribution.

The book is filled with interesting and most useful information. The vast cooperative effort which is required to accomplish successfully the many tasks of an undertaking of the importance of the Plutonium Project is somewhas reflected in the number of forewords, prefaces and introductory notes. This book contains seven, not counting the introductory chapter by the editor.

There is also some repetition, unevenness in length of chapters and extent of bibliographies noticeable throughout the book but that can hardly be avoided

in any cooperative work. Due to the somewhat historic presentation of the material, it is also difficult to bring all data up to date. Some values such as, for example, the permissible toferance exposures as used now are lower than the ones given in the book.

These few defects are small compared to the great value of the material presented. The book can be highly recommended both for its scientific content and its historic description of a great chapter in applied science.

—Otto Glasser, *Ph.D.*
Department of Biophysics
Cleveland Clinic

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OTHER LITERATURE

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to enable it to get the high-powered technical advice which would be unavailable otherwise.

The AEC, in part by edict of Congress and in part by its own doing, has operated with a plethora of advisory committees.

Now the Ad Hoc Advisory Committee on Cooperation between the Electric Power Industry and the AEC recommends strongly (see p. 78) the setting up of a permanent advisory group, representative of the power industry. This suggestion is based on the committee's conclusion that the prospects of nuclear power being economically significant within the next decades are "robust enough to warrant a strong present and continuing interest on the part of the electric power industry."

The primary objectives of a permanent group would be (1) to keep the industry up to date on important developments in reactor technology and (2) to advise AEC in areas where the power industry has had relevant experience.

There is no question but that with cooperation such a group could achieve its objectives. Both industry and the AEC program would benefit. However, before such a committee is set up, the contributions of other industries should be reviewed, e.g., the chemical, construction and metallurgical industries. Perhaps an advisory group from each of these should be set up. But surely doing this would make management of the program extremely cumbersome.

It would appear to us that, if another advisory committee is established, it would be much more logical that a committee representative of all industry be set up. This was in fact the recommendation of an industrial advisory group which reported to the AEC in 1948. —*J.D.L.*

Random Notes *(Continued from page 5)*

these cases, their subsequent use will be determined by how they compete in price with other apparatus. In this connection, it is expected that the AEC may shortly arrange to have a non-AEC organization do a cost analysis of the AEC isotope program.

• *The search for new sources of uranium in Canada has entered a new phase with the announcement by Eldorado Mining and Refining, Ltd., of its intention to go into production at its Ace Mine in the Goldfields of Saskatchewan. The new mill is to have a minimum capacity of 500 tons daily. It is expected that the output of the Ace Mine will equal or exceed that of Eldorado's original mine at Port Radium on Great Bear Lake. It can now be said that the treatment of uranium ores of the types generally found in Canada presents no greater difficulties than those encountered in the treatment of cobalt ores or arsenical gold ores.*

• The Atomic Energy Commission is considering the organization of a group which would handle proposals by industrial concerns and other organizations interested in participating in the atomic energy program at their own risk. This group would be affiliated with the General Manager's office.

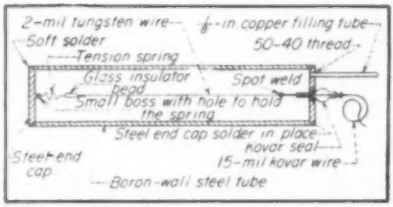
• *A recent survey of 41 industrial research laboratories shows the following methods of internal communication used by lab staff members (in order of decreasing frequency): personal visits, conferences, telephone, written memos. "Written memos" is last because it has been found that there is an inability to write and understand them.* —*J.D.L.*

CROSS SECTIONS

Rebuilding Boron-Wall Neutron Counters

By E. H. Daggett
Mound Laboratory, Miamisburg, Ohio

Most boron-wall tubes fail for either of two reasons: a broken center wire or end insulator, or a leak that has allowed air to enter the tube. In addition to these, there is the more remote possibility that the ethyl ether that is used as a quenching agent has been depleted by a very large number of counts. In any event, the most satisfactory repair is to rebuild the tube completely. Such a procedure is described here. This method of rebuilding tubes has been applied at Mound Laboratory to boron-wall tubes from the Argonne National Laboratory as well as to those that are made by the General Electric Company. This method has extended the life of the boron-wall shells indefinitely. The characteristics of the repaired tubes are as good as when the tubes were received new.



Modification of boron-wall counter to facilitate reconstruction

There are some modifications of the boron-wall tube that can be made in reconstruction so that less expensive and more readily obtainable seals may be used. As shown in the illustration, the end caps are made of cold-rolled steel; the tension-spring suspension is different from the original; and a ⅛-in. copper exhaust tube is used.

The old tube is taken apart by twisting and slipping off the brass shell, unsoldering the center wire from the high-voltage connector, unsoldering the ground wire, and removing the brass end from the steel tube by loosening the tangent screw that holds it in place. The end caps are removed by heating with a torch or large soldering iron, and the tube is cleaned out with ethyl ether.

A new center wire is made by spot-welding a 10¾-in. length of 2-mil tungsten wire to a 4½-in. length of 15-mil Kovar wire. The free end of the tungsten wire is forced into the molten end of a soft-glass insulator bead of about the same size as the original bead. The other end of the bead is heated, and the tension spring is forced into it. This spring is made from five turns of 1-mil wire wound on ⅛-in. diameter rod.

The tube is then clamped in a vertical position, bottom end up, and the new tinned end cap, with its spring, insulator and center wire, is soldered into place. It is very important to use a minimum of both solder and flux, to prevent them from running down inside the tube. These materials would not only cover up the wall and decrease the effective area of the boron, but the flux might outgas and make it difficult to get good evacuation.

A tinned, top end cap and Kovar seal are threaded over the 15-mil Kovar wire, and the wire is bent to prevent it from sliding back into the tube. The whole assembly is inverted so that the top end cap is up; the cap is then soldered into place along with the Kovar seal and exhaust tube.

The next operation is to apply tension to the center wire and to solder it into place. To do this, a spring similar to the internal spring is hooked onto the end of the Kovar wire, and tension is applied until the external spring is extended about ⅛ in. By this means, it is possible to know how much the internal spring has been extended. Solder is then flowed into the hole in the seal around the wire to secure it and to seal the tube.

The tube is now ready for evacuation and filling. The exhaust tube is waxed into a vacuum system capable of reaching a pressure down to at least 5×10^{-5} mm of mercury, and the steel tube should be outgassed by heating it with a torch. The filling mixture consists of purified ethyl ether (0.7 cm Hg), used as a quenching agent, and neon (8.8 cm Hg). The ether is purified by the addition of metallic sodium to its containing flask while on the vacuum system. Before the tube is removed from the vacuum system, it is convenient to use a scaler and a neutron source to check that the tube counts satisfactorily.

After the tube has been filled and checked, the exhaust tube is crimped shut in three places with a tool made by grinding out part of the jaws of a pair of diagonal cutting pliers. This crimping closes off the tube; it may then be cut from the system and the end dipped in a solder pot to ensure that the seal will not leak.

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*Colorimetric Method for Dosimetry of 10–100 r**

By G. V. Taplin, C. H. Douglas, and B. Sanchez
School of Medicine, University of California at Los Angeles
Los Angeles, California

A modification of the two-phase chloroform-dye indicator system of radiation measurement reported a year ago,† provides a suitable method for indicating doses of 10–100 r. The original system was designed to measure doses of gamma radiation at values of 200, 400, and 600 r. Acid evolution from chloroform was found to be proportional to radiation dose, and the *p*H of a bromcresol dye solution overlaying chloroform was adjusted so that the dye changed color at the desired point.

The increase in sensitivity is accomplished by removing or reducing the alcohol content in chloroform. The alcohol is added by the manufacturers as a preservative, and may be removed from reagent-grade analytical chloroform by washing it ten times with equal volumes of distilled water. After each period of shaking, the mixture is allowed to separate for 15 minutes in a large separatory funnel; the washed chloroform is removed and again washed in a similar manner. Samples prepared in this manner from the same brand and lot of the analytical-grade reagent have

* This article is based on work performed under contract AT-04-1-GEN-12 between the Atomic Energy Commission and the University of California at Los Angeles, as reported in the AEC document UCLA-118.

† G. V. Taplin, C. H. Douglas, reported in Nucleonics **6**, No. 6, 66 (1950); see also *Radiology* **56**, 577 (1951).

similar reactivities to X- or gamma radiation. However, there is a so far unexplained difference in radiation reactivity between different brands and lots of the same brand; this gross difference is being studied.

The alcohol-free chloroform-dye system has been shown to be 25–50 times more sensitive than the original, which contained approximately 0.5% ethyl alcohol. When 0.5% absolute ethyl alcohol is added to the alcohol-free system, the sensitivity returns to its original state.

Experiments

Before chemical and instrumentation specifications for the low-range system were developed, the system's reactivity to ultraviolet radiation, energy and temperature dependence, and reactivity to variations in radiation intensity were studied. In these studies, Mallinckrodt's analytical-reagent-grade chloroform was used. The bromeresol purple dye was obtained from National Aniline Co.

Dye solutions were prepared by adding 80 mg of the acid salt to 90 cm^3 of distilled water and heating the mixture almost to boiling to effect complete solution. This stock solution was adjusted to *p*H 5.0 by adding 0.01*N* NaOH; the volume was then made up to 100 cm^3. A 10:1 dilution of this stock solution was used in most of the experiments.

In the radiation studies, a 2:1 ratio of chloroform to dye was used. Total acid evolution following radiation of samples in 1-cm^3 stoppered volumetric Pyrex flasks was determined by titrating the sample with standardized 0.001*N* NaOH until the color of the dye phase returns to that of the dye solution that was originally overlayed on the chloroform.

A 250-kv 15-ma X-ray tube with inherent filtration equal to 0.21 mm Cu, and 1, 10, and 200 mg standard-

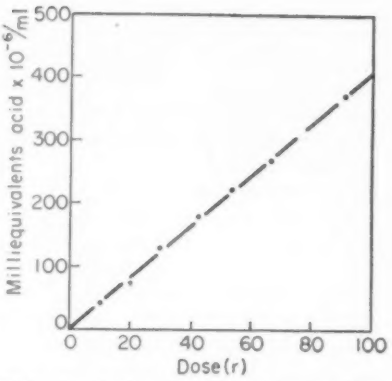


FIG. 1. Acid production from alcohol-free chloroform after exposure to gamma radiation in low dosage range

ized radium gamma-ray sources were used for irradiation of the samples.

Sensitivity to gamma irradiation. A linear relation between acid production from alcohol-free chloroform following gamma irradiation and dosage rate was found to exist from 0 to 750 r. Because the main use of the alcohol-free system is in the low dosage range (100 to 10 r or less), studies were conducted to make sure that there is no induction period or deviation from the linear relation when the same batch of chloroform is exposed to various doses below 100 r. Figure 1 shows the arithmetical mean of 21 determinations at each of 8 dosages between 10 and 100 r; acid production per roentgen remains constant.

Intensity and energy dependence. The acid production per roentgen was found to be dependent on the dosage rate and on the energy of the radiation to which the system was exposed. When exposed to radium gamma radiation, acid production varied from 2.90×10^{-6} milliequivalents/ml/r at 200 r/hr, to a maximum of 4.50×10^{-6} milliequivalents/ml/r at 20 r/hr.

X-ray studies showed that the system is more sensitive to low-energy radiation than to gamma rays from radium, showing a wave-length depend-

ence in the 0.26–0.015 A. U. range. However, by shielding the samples with 0.15–0.45 mm of lead, the effects of soft radiation may be greatly reduced without altering the absorption of radium gamma rays, thus minimizing the effects of wave-length dependence.

Heat stability. When alcohol-free chloroform is exposed to elevated temperatures (25 to 60° C), it decomposes rapidly, liberating large amounts of HCl, HOCl and phosgene. However, if overlayered with pure water or aqueous bromcresol purple and kept in flame-sealed Pyrex tubes, its heat stability is nearly as good as the alcohol-stabilized reagent. Under these conditions, the alcohol-free dye system is stable for at least six months at room temperature and for three months at 37° C. When the samples are exposed to temperatures between 70 and 80° C, which are above the boiling point of $CHCl_3$, decomposition takes place within 24 hours. The same system withstands repeated rapid freezing (liquid nitrogen) and thawing.

Light sensitivity. The dye solution itself is decolorized by prolonged exposure to direct sunlight. Both alcohol-free and alcohol-stabilized cholorform liberate acid upon short exposures to ultraviolet light (2,537 A. U.). The alcohol-free system is almost 10 times more sensitive than the stabilized system.

Vibration and jarring. Both systems have been sealed in small serum vials (neutra glass) and subjected to mechanical vibration (60 cycles) eight hours daily for five weeks without detectable color changes or differences in radiation sensitivity. Similar samples have been worn by personnel and strapped to the front axles of vehicles which were driven over the desert for 30 days. The only samples showing decomposition were those which also showed evidence of leakage around the plastic seals.

Instrumentation

Construction of a colorimetric dosimeter requires adjustment of the end point of the dye solution to the desired radiation dose, fabrication and filling of vials that hold the chemical reagents, and proper design of an external container to shield the dosimeter from light and soft radiation.

Adjustment of end point. There are several ways to adjust the system to measure specified doses of gamma radiation after the acid production/r of the particular batch of alcohol-free chloroform has been determined. The method adopted here is to add equivalent amounts of NaOH to the dye (*p*H 5.0), as determined from the acid production/r that is shown in Fig. 1. The dye concentration (1:10 dilution of stock solution) and the 2:1 ratio of chloroform to dye (1.0 and 0.5 cm^3) are kept constant. The amounts of NaOH added to the system discussed here are:

Dose (r)	*Milliequivalents* $\times 10^{-6}$ *NaOH* / *0.5 cm^3 dye*
10	41
25	102
50	204
100	408

Following radiation exposure, acid formed in the chloroform diffuses into the dye phase, reducing its *p*H and thereby producing the color reaction. Bromcresol purple solutions undergo a series of color changes when their *p*H is varied from 7.0 to 5.2. The dye is purple at *p*H values above 6.5, violet at 6.4–6.2, red at 6.1–5.9, amber-red to amber between 5.8–5.4, and yellow below 5.2. In spite of these intermediate color reactions, the yellow end point of this dye is sharp. For example, when the alcohol-dye system is adjusted to measure 10 r, radiation with 5–7 r causes no appreciable color change. After 8 r the dye becomes red-violet,

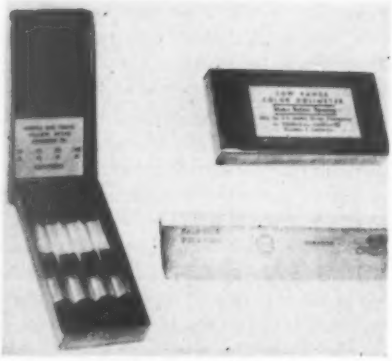


FIG. 2. Prototype model of low-range colorimetric dosimeter

at 9 r amber, and at 10 r yellow. Further radiation causes only slight changes in the shades of yellow. Thus, if the bright yellow color is selected as the final end point, dosage can be estimated with an accuracy of about 10–15%. For example, samples set to turn yellow from purple after specified doses begin to reach the yellow end point at doses about 10–15% less than these values.

Fabrication and filling of vials. The major problem in the instrumentation has been to find a satisfactory procedure for sealing the chemical reagents in hard-glass tubes. Flame sealing methods ordinarily used by pharmaceutical manufacturers are inadequate because acidic substances are produced from the decomposition of traces of chloroform or dye adhering to the surface of the glass at the site of sealing.

To obviate these difficulties, the following flame sealing procedure was developed:

1. Use Pyrex tubes, cleaned with chromic acid cleaning solution and rinsed repeatedly with distilled water.
2. Prepare tubes for filling by flaming and drawing out to form a narrow opening.
3. Add reagents through a hypodermic needle.
4. Immerse samples in dry ice and acetone until frozen.
5. Flush tubes with pure nitrogen gas, through a hypodermic needle.
6. Flame seal constricted end of glass tube, using a microject gas-oxygen flame.

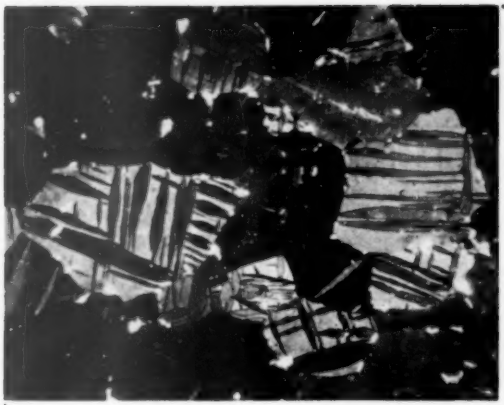
With this method, 70–80% of several thousand tubes have been sealed satisfactorily without introducing appreciable quantities of acid during the process. The accuracy of the sealed samples may be graded by color comparison. However, the crucial test is calibration by radiation exposure to a standardized radium source.

External containers. The cases for the low-range dosimeters shown in Fig. 2 were made of half-hard sheet copper 0.6 mm thick. The ends of the tubes were capped with hollow rubber stoppers to protect them from shock. The metal containers were designed to protect the chloroform-dye system from light, heat flash and from soft X- and gamma irradiation. For personnel use, the dimensions of the external and internal containers may be made smaller and the instruments could be redesigned for greater convenience in wearing or carrying.

Discussion

In our original alcohol-stabilized chloroform-dye system, the acid production following radiation given in a series of several short exposures was equal to that produced by a single exposure, when the total dosage was the same. For example, samples prepared to turn yellow at 400 r could be irradiated in 50- or 100-r steps or with a single 400-r exposure, and the color change would take place at approximately 400 r under either circumstance.

Similar experiments have been performed with the alcohol-free chloroform-dye system. Results are likewise additive. However, there is an important difference. During the first 10–15 minutes following irradiation, there is a definite after-reaction amounting to



BERYLLIUM, much used in reactors and neutron source construction, is difficult to fabricate. Contrary to properties exhibited by Be produced by more common methods, vacuum-sintered Be made by powder-metallurgy methods shows some ductility which permits cold rolling up to 10% reduction. Photomicrograph shows mechanical twin formation (dark strips on light background) during cold rolling; the twins disappear after annealing at 800° C, and grain growth starts. New technique is the result of AEC research carried out at the Metallurgical Laboratories of Sylvania Electric Products, Inc., Bayside, N. Y.

about 5–10% additional acid formation. This may be related partly to the time required for complete diffusion of acid into the aqueous phase. However, because this phenomenon has not been noted with alcohol-stabilized samples, it appears to be a continuing chemical reaction. It supports the theory that the mechanism of acid production in radiated chloroform involves a chain reaction. Alcohol may shorten the chains, thereby reducing the sensitivity of the water-saturated chloroform-dye system.

The alcohol-free system is sufficiently sensitive to measure gamma radiation in doses as low as 10 r, even when low chloroform-dye ratios and strong concentrations of dye are used. By reducing the concentration of dye and adjusting its *p*H closer to the yellow end point and by using higher chloroform-dye ratios, doses of 1 r or less may be detected colorimetrically. For field use, this degree of sensitivity of the method is not practical at present. However, there are probably many important industrial and laboratory applications for the method in measuring accumulative dosage in the range between 1–10 r. From the data presented, the alcohol-free system is readily adaptable to measure doses of residual gamma radiation from a nominal atomic bomb explosion between 10–100 r (the emergency military tolerance range). However, the factors of wave-length and temperature dependence require consideration.

It has been shown that small amounts of ethyl alcohol increase the stability of the system to heat and ultraviolet light and reduce the temperature dependence. Further studies are needed to determine the optimum percentage of alcohol which could be used in the system for added stability, at the same time allowing sufficient sensitivity to measure the low dosage range (10–100 r). This optimum ethyl alcohol concentration lies between 0.02 and 0.20% by volume.

* * *

We gratefully acknowledge the help of Drs. B. Cassen, M. Greefield, S. Wilcher, T. J. Haley, F. A. Bryan, N. MacDonald, and Mr. N. Todd.

NUCLEONIC EVENTS

POWER GROUP RECOMMENDS INDUSTRY-AEC COOPERATION

Prospects of nuclear power becoming significant in our economy during the next decades "are robust enough to warrant a strong present and continuing interest on the part of the electric power industry." This is the most significant conclusion of the recently released report of the Ad Hoc Advisory Committee on Cooperation between the Electric Power Industry and the AEC. Electric power capacity, according to the report, "has doubled on the average every decade for the past forty years, and it is reasonable to believe that this will happen again in the next ten years, and that a substantial rate of growth will continue thereafter."

Therefore, the industry should be strongly attracted by any system "which might bring about a material lowering in cost of steam power production." And "apart from the possibility of cost reduction through nuclear reactors, such a new source of heat energy might afford advantages in compactness relative to conventional fuel and in the extensive supplies that would become available if breeding is perfected."

In addition to the power industry's interest in the AEC program, the committee observed that there is much in the industry's experience which could be used by AEC. Common problems include such things as the design of pressure vessels, of coolant systems (and control of purity of coolants), of pumps, piping, and heat-transfer systems; the electrical, mechanical and civil engineering aspects of reactor plant construction; fuel preparation and ash handling; control and instrumentation; and the general relationship between reactor operating characteristics and those of conventional electric power plants.

More important, the committee feels, are "realistic appraisals of the potentialities of reactor projects as parts of electric power systems." "In pursuing the idea of a nuclear reactor for the production of plutonium and by-product heat-energy for power," the application of utility economics becomes "pointedly relevant."

"The problem will become increasingly complex and pressing as the time approaches when a nuclear reactor is designed primarily for the production of heat energy for a power plant. The Commission then will require more and more refined analyses of such important items as: capital costs of reactors and their complementary parts to give total investment costs in a complete system of electric power generation; dependability of reactors, that is the proportion of the time that the reactors would be reliably available for power supply at full capability; costs of reactor maintenance; costs in starting and stopping units; costs of feed material and handling of feed material, including prospects as to cost and availability of such fuel at least covering the period of the economic life of the initial investment; the problems of integration of nuclear power into existing power networks; and finally the over-all costs, both capital and operating, of the power obtained from such nuclear reactors and the relative advantages or disadvantages of such power as compared

with that from other competing sources for which, if economical, it would be substituted."

Committee Recommendations

Because of the important mutual interests which the AEC and the electric power industry have, the committee recommended that permanent cooperation between the two groups be set up. The mechanism for establishing this cooperation would be by means of an industry advisory group consisting of ten to fifteen representatives of the power industry. The functions of this group would be to:

1. Provide organized power industry assistance to the various AEC projects in identifying places where industry personnel could be helpful and to locate personnel within the industry who might be detailed to fulfill these needs.
2. Familiarize itself with the AEC program and make reports to the power industry to keep it abreast of developments in the atomic energy field and of their implications.

In connection with the first function listed, the committee estimates that approximately 25 technicians per year could be used effectively on a temporary basis by the AEC on problems where electric-industry experience is relevant.

As they return to their own companies, the men assigned to these jobs would form a reservoir of informed personnel for the industry. Further along these lines, the committee recommends that the industry send employees to the Oak Ridge School of Reactor Technology.

The members of the Ad Hoc Committee were: Philip Sporn, president, American Gas and Electric Co., chairman; E. W. Morehouse, General Public Utilities Corp.; and Walton Seymour, adviser on power problems, Economic Cooperation Administration.

OAK RIDGE SYMPOSIUM TO HEAR GLENNAN, HAFSTAD, AND SUITS

Participants in the 1951 Oak Ridge Summer Symposium which will be held in Oak Ridge, Tenn., from August 27 to September 7, will hear talks of general interest by T. Keith Glennan, Lawrence R. Hafstad, and C. G. Suits. The symposium has as its theme "The Role of the Engineer in Nuclear Development" (NU, June '51, p. 43) and will include talks on technical phases of the subject by nineteen other engineers, educators, and physicists.

Mr. Glennan, member of the Atomic Energy Commission, will discuss engineering and the objectives of the AEC, while Dr. Hafstad, who is director of the AEC's Division of Reactor development, will discuss the AEC reactor program. Dr. Suits, vice president and director of research of the General Electric Company, will speak on some of the economic aspects of atomic power.

ARCHITECTS PLAN CONFERENCE ON "HOT"-LAB PROBLEMS

The American Institute of Architects, with the Atomic Energy Commission and the Building Research Advisory Board of the National Research Council as co-sponsors, is planning a conference in October to discuss building problems connected with the utilization of nuclear energy in laboratories and hospitals. The program will be devoted entirely to better building construction for peacetime uses of nuclear energy.

Subjects to be discussed include radiation shielding, waste disposal, air supply and ventilation for hoods, building materials and finishes not easily contaminated, and architectural designs for tracer laboratories.

The meetings will be held at the National Research Council. Tentative dates are October 22 and 23. Persons interested in attending can

contact William H. Scheick, Executive Director, Building Research Advisory Board, National Research Council, 2101 Constitution Avenue, Washington, D. C.

AEC MAKES 73 MORE ISOTOPES AVAILABLE FOR EXPORT

Seventy-three radioisotopes have been added to the list of those available for foreign export, the Atomic Energy Commission announced last month. In enlarging its radioisotope export program, the AEC has included all radioactive materials now sold in this country on an unrestricted basis, and for the first time made U.S.-produced radioisotopes available to foreign users for industrial research and applications.

The action increases the number of U.S.-produced isotopes available to buyers in foreign countries from 26 to 99. Among the more useful of the newly-available isotopes are cesium-137, yttrium-91, selenium-75 and tantalum-182, which all have valuable applications in industrial research; chromium-51, nickel-59 and 63, and tungsten-185, which are useful in metallurgical research, and rubidium-86, which is a valuable substitute for the shorter-lived sodium-24 and potassium-42 in agricultural research.

One isotope distributed in the U. S. on a limited basis—hydrogen-3 (tritium)—is not available for export.

The "very high degree of success with which the program has been operated in the past" and the benefits derived from the use of isotopes throughout the world were given by the Commission as the reasons for the program's expansion. The action, in addition, provides permission, for the first time, for American manufacturers to export radioactive thickness gages, which are finding increasing application in a number of different industrial processes.

The new isotope program also permits the Oak Ridge National Laboratory to provide special irradiation services to scientists of other nations. Under this program, foreign scientists may send special materials to Oak Ridge to be made radioactive by neutron irradiation in the reactor there.

If and when the demand for any particular U.S.-produced isotope exceeds the supply, no foreign orders will be filled until all domestic applications have been taken care of.

The procedures which foreign buyers must use in ordering isotopes from the U. S., as well as information on the procedures U. S. buyers must use in ordering isotopes from Canada and the United Kingdom, are described in the 1951 Isotopes Catalog, available from the AEC Isotopes Division, Export-Import Branch, Oak Ridge, Tenn.

The radioisotope export program has been in effect since September 3, 1947. Since then 34 countries have completed arrangements to receive shipments.

80 SYMPOSIA, 900 PAPERS PLANNED FOR CHEM CONCLAVE

The final program for the World Chemical Conclave to be held in New York and Washington from September 3–15 was recently announced, with 80 symposia and more than 900 papers scheduled for presentation.

The Conclave includes the 75th Annual (Diamond Jubilee) Meeting of the American Chemical Society (New York, September 3–7), the 16th Conference of the International Union of Pure and Applied Chemistry (New York, September 8–9; Washington, September 14–15), and the 12th International Congress of Pure and Applied Chemistry (New York, September 10–13).

Radioactivity, as related to the various branches of chemical research, is the subject of numerous papers on the program. Of particular interest to

NUCLEONICS' readers will be, for example, the six papers scheduled for the ACS symposium on nucleonics and tracer techniques in analytical chemistry on September 4, three papers to be presented as part of the ACS symposium on liquid industrial wastes on September 7, and the 50 papers on topics related to nuclear chemistry that are scheduled as part of the International Congress, September 10–13.

NEW URANIUM MINE OPENS IN CANADA

The Eldorado Mining and Refining Company has started developing the uranium deposits at Beaver Lodge Lake in Saskatchewan which are expected to yield four to five times as much ore as the Port Radium mine on Great Bear Lake, previously the largest Canadian uranium producer.

In the Beaver Lodge district, whose uranium deposits have been described as the biggest yet discovered on the North American continent, pitchblende has been found 900 feet below the surface. At Port Radium, the mining is done at about 1,300 feet.

It is expected that the minimum daily output at Beaver will be 500 tons, with the possibility of doubling or tripling this figure in a short time. A mill under construction will have a capacity of 500 tons and is designed for expansion to a 2,000-ton capacity.

The mill will also be operated by Eldorado, which is a company owned by the Canadian government. Ores taken from other mines near the Eldorado property will be processed at the Eldorado mill. Construction of mills is contemplated in other areas where good ores are available from two or more mines but where individual tonnages will not support a mill.

Canada's uranium requirements are not large, and even the needs of the proposed additional reactor development at Chalk River will not tax the new resources. At present, the United States is the only export market for Canadian uranium.

ORINS COUNCIL ELECTS THREE NEW SPONSORS

Election of three new sponsoring institutions for the Oak Ridge Institute of Nuclear Studies was part of the business conducted by ORINS' Council at its recent annual meeting. The institutions, which include Florida State University, the University of Maryland, and the University of Puerto Rico, bring to 29 the number of universities that sponsor the nonprofit educational corporation.

The Council also elected William V. Houston, president of Rice Institute, to a three-year term on the Board of Directors, which administers the affairs of the Institute in behalf of the Council. He succeeds Harold W. Stoke of the National Citizens Committee for the Public Schools. Paul M. Gross, vice president of Duke University, and Jesse W. Beams, professor of physics at the University of Virginia, were re-elected to three-year terms on the Board.

At a meeting of the Board of Directors which followed the Council meeting, Dr. Gross was re-elected president of the Institute and Dr. Beams was re-elected vice president.

BRITISH LORDS SEEK NEW ORGANIZATION OF A-PROJECTS

Following on the heels of the news that Lord Portal, Great Britain's Controller of Atomic Energy, and Michael Perrin, his principal scientific adviser (see page 86), had resigned, the House of Lords on July 5 passed a resolution by a vote of 52-31 criticizing the slow progress made "in developing atomic energy for peaceful and for warlike purposes" and urging the government to transfer the program from the

Ministry of Supply to a special organization directly under the Prime Minister.

Lord Cherwell, who, as Professor Lindemann, served as wartime scientific adviser to Winston Churchill, pressed the matter to a vote by pointing out that both Lord Portal and Mr. Perrin felt a reorganization is necessary. He said that persons working on the program are receiving much less than private firms would pay and that a large proportion of the best men had already resigned or were trying to leave.

Attacking the security setup and criticizing the government for not only failing to keep pace with the United States but falling far behind Russia in the development of an atomic bomb of its own, Lord Cherwell charged that Britain's atomic projects are so hamstrung by the Civil Service that they are "threatened with collapse and disintegration."

Lord Alexander, in reply, upheld the existing system and praised the power reactor design work being done and the large production facilities that have been established in England.

AUSTRALIA'S ACCELERATOR NEARING COMPLETION

Professor E. W. Titterton announced recently that Australia's 1-Mev cyclosynchrotron, under construction at Canberra, will be ready this year. Speaking before the Brisbane Congress of the Australian and New Zealand Association for the Advancement of Science, he told of the work of Marcus Oliphant who designed the instrument and is supervising its construction.

Professor Titterton, who for the past four years has headed up a nuclear research team at the Atomic Energy Research[7] Establishment at Harwell, England, has just been appointed to the chair of experimental physics at Canberra National University.

AEC SANTA FE OFFICE MAKES PERSONNEL CHANGES

In preparation for the transfer of AEC's Santa Fe Operations headquarters from Los Alamos to Albuquerque this month, personnel changes and assignments have been made as follows:

George P. Kraker, former Sandia Field manager for AEC at Albuquerque, is deputy manager, Santa Fe Operations. He will assist C. L. Tyler, manager of Santa Fe Operations for AEC.

Daniel F. Worth, Jr., formerly Mr. Kraker's assistant, succeeds him as Sandia Field manager. In this capacity he represents the AEC in administering contracts with the Sandia Corporation, the Armed Forces Special Weapons Project, and the Air Force's Special Weapons Command at Kirtland Field, Albuquerque.

Elmo R. Morgan, who was assistant to the manager of Santa Fe Operations and director of Los Alamos community management, has been made manager of the new Los Alamos field office. He will direct all AEC activities at Los Alamos, including administration of various research, community operations and other contracts.

The transfer of all units of the Santa Fe Operations office to Albuquerque is not expected to be concluded until about November 1 of this year.

ARGONNE REACTOR BUILDING BIDS OPENED

Bids for the construction of a building to house a new research reactor at Argonne National Laboratory have been opened and work will begin 10 days after the name of the successful bidder is announced, which is expected at an early date.

The new building is to be erected close to the other facilities of the laboratory at its Du Page County site, 30 miles southwest of Chicago. Comple-

tion of the structure is expected in twelve months.

The circular main structure of the building will be air-conditioned. Construction will include installation of building services and mechanical and electrical equipment to insure a compact, completely contained research reactor unit.

The reactor itself will be a modernized version of the heavy-water reactor now in operation at the laboratory's Palos Park site. The design is expected to provide for unusual flexibility in operation. When the new reactor is completed the Palos Park reactor will be dismantled.

AEC CREATES DIVISION OF CONSTRUCTION AND SUPPLY

The Atomic Energy Commission, in announcing recently that it had established a Division of Construction and Supply, said that the Division would be responsible for staff supervision of Commission construction and related engineering activities. It will also administer priorities, allocations and the Controlled Materials Plan for the AEC, and handle the production facilities licensing and export control programs.

Edward J. Bloch, former deputy director of the Division of Production, has been appointed director of the new Division, Frederick H. Warren, deputy director, and John R. Brindel, assistant to the director. Neil J. Carothers, Jr., has succeeded Mr. Bloch as deputy director, Division of Production, and James L. Kelehan has been appointed assistant director, Division of Production.

The Division will consist of the Construction Engineering Branch, under William K. Maher, the Defense Requirements Branch under Eber R. Price, the Export Control Branch under Lyall E. Johnson, the Supply Branch under George C. Taylor, and the Special Assistance Branch under C. R. Lee, Jr.

RESEARCHERS GET 15 NEW CONTRACTS FROM AEC

Fifteen new unclassified research contracts in physical and biological and medical research were awarded by the Atomic Energy Commission during the month of May. The project titles and the recipients of the contracts are as follows:

In chemistry: Catholic University of America (F. O. Rice), *Thermal production and identification of free radicals;* and Northwestern University (James N. Pitts, Jr.), *Investigation of the photochemistry of organic acids, ethers, and ketones.*

In metallurgy: Columbia University (C. Bonilla), *Boiling and condensing of liquid metals;* and Graham, Crowley and Associates (C. A. Crowley), *Electrowinning of zirconium.*

In physics: Princeton University (Lyman Spitzer), *Transport phenomena in ionized gases.*

In biology and medicine: Johns Hopkins University (Curt P. Richter),

Reprints Available

Reprints of the 32-page special report on "Practical Aspects of Radioactivity Instruments" published in June, and the 84-page "Guide for Selection of Equipment for Radioactivity Laboratories," including the 1950 Buyers' Guide, are available at 50 cents each. Also available are a limited number of reprints, at 15 cents each, of the three articles detailing the recommendations of the International Commission on Radiological Units and the International Commission on Radiological Protection. Your order and remittance should be mailed to NUCLEONICS, Editorial Department, 330 W. 42nd St., New York 18, N. Y.

Study of the part played by adrenals in the ability of rats to withstand radiation effects; Rutgers College (James E. Gunckel), *Histological and physiological effects of irradiation of* Tradescantia paludosa; University of Illinois (I. C. Gunsalus), *Metabolic pathways in microorganisms;* University of Nebraska (E. F. Frolik and R. Morris), *Genetic effects of thermal neutron irradiation of crop seeds;* Johns Hopkins University (Thomas G. Ward), *Metabolism of phosphorus in virus-host systems;* Johns Hopkins University (Jonas S. Friedenwald), *Enzymatic histochemistry of the ocular lens;* Smithsonian Institution (Robert B. Withrow), *Biochemical investigation of photomorphogenesis in green plants;* Harvard University (D. G. Cogan and D. D. Donaldson), *Sterophotography of anterior segment of eye with special reference to crystalline lens;* Columbia University (P. G. Hudson and J. M. Reiner), *Turnover of specific proteins, protein fractions, and nucleic acids, in normal and malignant human testis and kidney;* and University of Chicago (W. H. Taliaferro), *Effect of localized X-irradiation on antibody formation and antigen localization.*

The new awards bring to a total of 409 the number of unclassified research projects supported by the AEC. Of these, 244 are in biology and medicine, and 165 are in physical research. In addition, the AEC supports 73 unclassified projects jointly with the Office of Naval Research.

"PHYSICS TODAY" THEME OF AIP SYMPOSIUM

An all-day symposium, with papers on nuclear, atomic, and solid-state physics, acoustics, and optics, is featured on the program arranged for the Twentieth Anniversary of the American Institute of Physics which will be celebrated at the Hotel Sherman in Chicago, Ill., October 23–27, by a joint meeting of the Founding Societies of the Institute.

The symposium, formed around the theme "physics today," will be held on Thursday, October 25 at the Chicago Civic Opera House.

The program lists papers to be given by Enrico Fermi, Nobel Laureate in physics who is now at th University of Chicago; E. U. Condon, director of the National Bureau of Standards; J. C. Slater, head of the Physics Department of Massachusetts Institute of Technology; Harvey Fletcher, of Columbia University; Edwin H. Land, president of the Polaroid Corporation; and K. K. Darrow, of the Bell Telephone Laboratories.

Senator Brien McMahon and Karl Compton will be guest speakers at a joint banquet on Thursday evening, October 25.

IN BRIEF

➤**Security standards** and "procedures applicable to the protection of atomic information" were discussed last month in London by high-level American, British, and Canadian representatives in a closed meeting concerning tightening of security measures.

➤***Amalgamation*** *of the technical information and report sections of the three military services became a reality on July 1 with issuance of a Department of Defense directive setting up an Armed Services Technical Information Agency (ASTIA). This new agency, to be concerned only with internal dissemination of information, will take over the work of the Air Force's Central Air Documentation Office at Wright-Patterson Field, the Navy's technical reports section operating from the Library of Congress, and the scattered Army operations. Businessmen and others will still have to obtain research reports from the Office of*

Technical Services of the Department of Commerce.

➤**Iodine-131** is being flown weekly to Argentina from the United States to aid in work leading to treatment of endemic goiter. The shipments are going to a ten-man research team from the Johnson Institute of the Harvard University Medical School who are working on a project of ten weeks' duration at the National University of Cuyo in Mendoza.

➤***Lithium,*** *discovered in rich deposits in the area, will be processed in a new pilot plant to be built in Manono, a Belgian Congo village. Geomine, the Belgian tin mining company, will operate the plant in accordance with an agreement with the United States government. Plans are already under way for construction of a larger, permanent plant.*

➤**Less paper work** for the AEC and its suppliers resulted from the issuance of Direction 1 to National Production Authority Regulation 2 recently. The direction calls for automatic conversion of outstanding rated orders for noncontrolled materials placed under the construction, operations, and privately owned facilities programs of the AEC to orders bearing the new rating and allotment symbols required by CMP Regulation 1. Orders affected were those calling for delivery after June 30.

➤***Pollution control*** *is the basis for U. S. Public Health Service stream sanitation studies of the Columbia River and its tributaries between Priest Rapids above the Hanford Plutonium Works project and Paterson below McNary Dam. Begun last month with a staff of 14 persons based in Richland, Wash., the field work is expected to take two years to complete. These studies are expected to supplement the stream studies which are carried out continually by the General Electric Company and the Atomic Energy Commission in connection with Hanford's operation.*

➤**The Atomic Scientists Association** of England has closed its offices at 7 Victoria St. in London. Future correspondence should be addressed in care of Prof. J. Rotblat, Physics Department, Medical College of St. Bartholomew's Hospital, Charterhouse Square, London, E. C. 1.

NUCLEAR NEWSMAKERS

Robert D. Huntoon, formerly chief of the Atomic and Radiation Physics Division of the National Bureau of Standards, has been appointed associate director of the Bureau. He will be in charge of the Bureau's recently established Corona Laboratories near Corona, Calif., where various phases of electronic research, development, and engineering are to be carried out.

Michael Amrine recently took up new duties as managing editor of the *Bulletin of the Atomic Scientists.* Mr. Amrine has written on many phases of nuclear science and was at one time in charge of public information at Brookhaven National Laboratory.

Lyle B. Borst resigned his post as chairman of the Department of Reactor Science and Engineering of Brookhaven National Laboratory on August 1. Dr. Borst joined Brookhaven in 1946 to take charge of the design, execution, and operation of its reactor and hot laboratory. With the completion of this assignment, he is returning to academic circles to take up teaching and fundamental physical research and has accepted a professorship in physics at the University of Utah.

Serge A. Korff, of New York University College of Engineering, recently returned from South America. He delivered a paper on Geiger counters at the Fifth South American Congress of Chemistry held in Lima, Peru, and lectured at the University of Chile in

Santiago, where he was elected an honorary member of the faculty.

Francis G. Slack recently resigned as chairman of the Department of Physics at Vanderbilt University. One of the original organizers and incorporators of the Oak Ridge Institute of Nuclear Studies, Dr. Slack will live in Florida where he plans to devote his time to writing and research.

Michael Perrin, senior researcher at the British Ministry of Supply's atomic energy project since its inception, has resigned his post to return to Imperial Chemical Industries.

Karl T. Compton, Lewis L. Strauss, Charles D. Coryell, and **Wendell C. Peacock** have been named to the newly created Research Advisory Board of Tracerlab, Inc. Dr. Compton, who is chairman of the new group, and Mr. Strauss, former member of the Atomic Energy Commission, are directors of Tracerlab.

Richard F. Humphreys, former assistant chairman, has been named chairman of the physics department at Armour Research Foundation of Illinois Institute of Technology. In this position, he will head research activities in nucleonics, light and optics, physics of solids, acoustics, high pressure, and electricity and magnetism.

Melvin Fields has been promoted to the position of associate technical director of Tracerlab, Inc., and appointed to its management staff. He formerly was director of the company's chemical division.

Dana W. Atchley, Jr., who resigned this spring as director of engineering of Tracerlab, Inc., has been appointed coordinator of technical research of United Paramount Theatres, Inc.

Carroll L. Wilson will direct the work of the newly formed Industrial Development Department of the Climax Molybdenum Company. He was formerly general manager of the Atomic Energy Commission.

Betty Cooper has taken a position at Oak Ridge where she will serve as part-time anesthesiologist with the Oak Ridge Hospital and as senior scientist with the Medical Division of the Oak Ridge Institute of Nuclear Studies. Dr. Cooper was formerly associated with the Department of Anesthesiology of the University of Texas.

W. C. Funk has been named deputy manager of the Atomic Energy Commission's Idaho Operations Office, where he has served as assistant manager since 1949.

John H. Van Vleck, Hollis professor of mathematics and natural philosophy, was recently appointed dean of applied sciences on the Harvard University faculty of arts and sciences.

D. R. Shoults, of St. Louis, Mo., will direct the General Electric Company's Aircraft Nuclear Propulsion Project for the Air Force and Atomic Energy Commission. Headquarters for the project is at G-E in Lockland, O.

William H. Sullivan reported on August 1 at Oak Ridge to take up his new post as chief research chemist in the Research Director's Division at Oak Ridge National Laboratory. Dr. Sullivan recently resigned his position as scientific director of the Naval Radiological Defense Laboratory in San Francisco.

George Granger Brown, director of the AEC's division of engineering, has accepted appointment as dean of the college of engineering of the University of Michigan. He has been on the faculty of the university since 1920.

James Franck, professor emeritus of physical chemistry in the University of Chicago's Institute of Radiobiology and Biophysics, last month received the

Max Planck medal, highest award of the German physical society. Dr. Franck, Nobel prize winner in physics in 1926, is currently engaged in research into the mechanism of photosynthesis.

Marion T. Clark, assistant professor of chemistry at Emory University, has been appointed assistant to the chairman of the university relations division of the Oak Ridge Institute of Nuclear Studies.

H. Walter Jones and **Russell E. Duff** have joined the staff of the Los Alamos Scientific Laboratory. Dr. Jones was previously employed as a chemist with the Western Regional Research Laboratory of the U. S. Department of Agriculture in Albany, Calif., and Dr. Duff was associated with the Engineering Research Institute at Ann Arbor, Mich.

George O. Curme, Jr., has been elected vice president in charge of research of Union Carbide and Carbon Corporation. Since 1948, he has been vice president in charge of chemical research for the Corporation and for many years a vice president of two of the Corporation's divisions, the Bakelite Company and Carbide and Carbon Chemicals Company, which operates the Oak Ridge National Laboratory.

MEETINGS

Oak Ridge Summer Symposium (on Nuclear Engineering) sponsored by Oak Ridge National Laboratory and Oak Ridge Institute of Nuclear Studies—Oak Ridge, Tenn., Aug. 27–Sept. 6

Low Temperature Physics Conference, UNESCO Commission for Very Low Temperatures—Oxford, England, Aug. 22–28; Institute International du Froid—London, England, Aug. 29–Sept. 5

American Chemical Society, Diamond Jubilee Meeting—New York, Sept. 3–7

American Mathematical Society, 472d Meeting—Minneapolis, Minn., Sept. 4–7

Twelfth International Congress of Pure and Applied Chemistry—New York, Sept. 10–14

Instrument Society of America, Sixth National Exhibit and Technical Meeting—Coliseum, Houston, Texas, Sept. 10–14

Institution of Mechanical Engineers and American Society of Mechanical Engineers, general discussion on heat transmission—London, England, Sept. 11–13

Biological Photographic Association, Inc.—Kenmore Hotel, Boston, Mass., Sept. 12–14

International Union of Chemistry, 16th Conference—New York, Sept. 8–9; Washington, D. C., Sept. 14–15

American Institute of Chemical Engineers—Rochester, N. Y., Sept. 16–19

International Conference on Nuclear Physics and Fundamental Particles—University of Chicago, Chicago, Ill., Sept. 17–20

Conference on Administration of Research—University of Michigan, Ann Arbor, Mich., Sept. 24–26

Colloquium on Sensitive Emulsions and Their Physical and Physical-Chemical Properties (sponsored by University of Paris, Faculté des Sciences de Paris, and the Centre National de la Recherche Scientifique)—Paris, France, Sept. 24–29

American Roentgen Ray Society—Shoreham Hotel, Washington, D. C., Sept. 25–28

American Society of Mechanical Engineers, Fall Meeting—Hotel Radisson, Minneapolis, Minn., Sept. 25–28

Fourth Conference on Gaseous Electronics (sponsored by APS Division of Electron Physics)—Schenectady, N. Y., Oct. 4–6

American Physical Society (New York State Section)—Rochester, N. Y., Oct. 5–6

39th National Safety Congress & Exposition, National Safety Council—Chicago, Ill., Oct. 8–12

World Metallurgical Congress (sponsored by American Society for Metals)—Detroit, Mich., Oct. 14–19

American Society for Metals, National Metal Congress and Exposition—Fair Grounds, Detroit, Mich., Oct. 15–19

Conference on Building Problems in Connection with Utilization of Nuclear Energy (arranged by American Institute of Architects, sponsored by AEC and NRC)—National Research Council, Washington, D. C., Oct. 22–23

National Electronics Conference—Chicago, Ill., Oct. 22–24

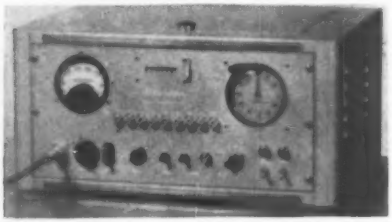
American Institute of Physics in joint meeting with Founding Societies of the Institute (American Physical Society; Optical Society of America; Acoustical Society of America; Society of Rheology; American Association of Physics Teachers; also American Crystallographic Association)—Hotel Sherman, Chicago, Ill., Oct. 23–27

American Physical Society in joint meeting with American Institute of Physics—Chicago, Ill., Oct. 25–27

Institute of Mathematical Statistics—Washington, D. C., Oct. 26–27

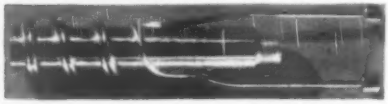
American Mathematical Society—Washington, D. C., Oct. 27

PRODUCTS and MATERIALS



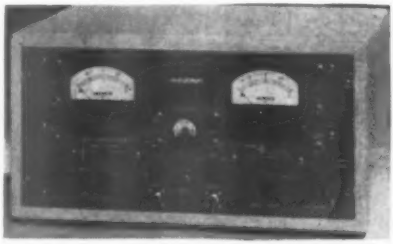
BINARY SCALER

Radiation Instrument Development Laboratory, 2337 W. 67th St., Chicago 36, Ill. The model 512 scale-of-512 scaler makes use of the Higinbotham scale-of-two circuit. Input sensitivity is variable from 10 mv to 20 volts, allowing use of scintillation and flow counters as well as G-M counters. Resolving time is said to be less than 5 μsec; register speed is 17 cps for evenly spaced counts. High voltage is continuously variable in the range 500–5,000 volts with electronic regulation of 0.01% of the change in line voltage.



HIGH-TEMPERATURE COUNTER

Radiation Counter Laboratories, Inc., 1844 W. 21st St., Chicago 8, Ill. This G-M counter (mark 1, model 15X) is filled with a self-quenching mixture of argon, xenon, oxygen, and nitrogen, and is designed to be operated in regions of high ambient temperature. The counter is maintained at room temperature by a $\frac{3}{32}$-in. stream of water flowing through the copper tubing that is soldered to the stainless-steel wall. When operated at an ambient temperature of 142° C the counter showed no detectable difference in characteristics from room temperature. The threshold is about 1,500 volts, plateau about 150 volts, and plateau slope 3–10% per 100 volts. Background is said to be about 50 cpm. Counters filled with this mixture are claimed to have counted more than 10^{10} counts without essential change in characteristics.



ANTICOINCIDENCE ANALYZER

Atomic Instrument Co., 84 Massachusetts Ave., Cambridge 39, Mass. The model 503B analyzer has been designed for counting extremely weak activities. Used with the large Libby screen-wall counter it permits the use of the large samples which low activities make necessary. The sensitivity of the transmit-amplifier channel is such that a pulse with a minimum amplitude of 0.5 volts at input will produce an output pulse of about 90 volts; the reject channel permits an input pulse of 0.25 volts, or more, to reject any pulse that comes from the transmit channel for 50–100 μsec after the reject pulse, independent of the pulse amplitude (0.5–100 volts) at the transmit-

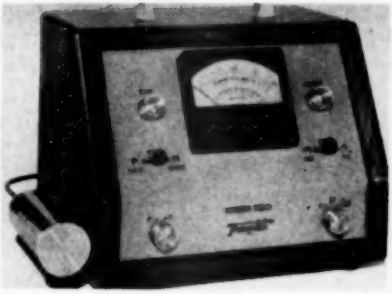
channel input. The unit comes with two line and load regulated 500 to 2,000-volt supplies with separate fine and coarse controls. A six digit register capable of registering twelve evenly spaced counts per second is also provided.

RATEMETER

Victoreen Instrument Co., 5806 Hough Ave., Cleveland 14, O. The model 524 ratemeter is designed for detecting and measuring alpha, beta, and gamma radiation. Six sensitivity ranges are provided, with full-scale readings from 300 to 100,000 cpm. The unit also includes a three-position time-constant selector and an enclosed loudspeaker. Provision for a scintillation counter as well as adaptability for various specialized counter tubes are incorporated.

URANIUM TEST KIT

Menlo Research Laboratory, Menlo Park, Cal. This pocket-size kit is for use with any type of ultraviolet instrument for on-the-spot identification of radioactive ore. The kit contains packages of testing chemicals, a 2,000° F blow-torch, bead-forming wires, and tongs for holding beads. All elements are standard and individually replaceable. From 25 to 30 bead tests can be made with a single kit.



MONITOR

Tracerlab, Inc., 130 High St., Boston 10, Mass. The model SU-3B laboratory monitor is for use as a routine monitor in radioactivity laboratories. It will detect low-energy beta emitters such as C^{14} and S^{35}. Equipped with a mica end-window G-M tube enclosed in a detachable probe assembly, the unit has three full-scale meter ranges of 200, 2,000, and 20,000 cpm. A volume adjustment is provided for the built-in loudspeaker. A built-in pulse generating circuit, synchronized with the 60-cycle line frequency, allows calibration checking without use of an external generator.

ANALOG DIGITAL CONVERTER

Genisco Inc., 2235 Federal Ave., Los Angeles 64, Cal. This device converts variable mechanical positions, such as the rotary positions of lead screws and servo motors, into electrical contact settings. Each standard decade unit has ten possible positions; a stack of three decades covers the number range from 000 to 999. The output is suitable for such uses as feeding information automatically to digital calculators or for operating electric typewriters. The

traversing speed is limited to speeds of the order of 250 number changes per second in either direction, or 2,500 with a special adapter. This limitation is a safeguard against brush wear; excessive speed itself would not produce errors because the device doesn't work by counting or accumulating pulses.

EPUT METER

Berkeley Scientific Corp., Sixth and Nevin Aves., Richmond, Cal. The events-per-unit-time meter (model 554) is a high-speed electronic counter combined with an accurate time base to provide an instrument that will automatically count and display the number of events that occur during a precise time interval. The instrument will count in the range 20–100,000 events per sec; these events may occur either regularly or with random distribution. It is claimed that short-term stability is better than 1 part in 10^5 and counting accuracy is ± 1 event. Results are displayed in digital form and no interpolation is necessary.

ORGANIZATIONAL CHANGE

Technical Associates, 3730 San Fernando Rd., Glendale, Cal. has purchased a substantial interest in the **Landsverk Electrometer Co., Cincinnati, Ohio.** The former will assume much of the responsibility for financial and business management; the latter will concentrate on development and production of quartz-fiber instruments. The name of Landsverk will be retained, but all business will be carried on from the California address.

POWER SUPPLY

Pedersen Electronics, Lafayette, Cal. The model 300 regulated power supply provides four outputs. The first supplies 250–300 volts at 0–300 ma: regulation is said to be better than 0.07% with load, 0.02% with line. Source impedance is less than 1 ohm from d-c to 40 kc, rising to 3 ohms at 100 kc. It is claimed that drift is less than 0.5% from a cold start, most of which occurs during the first hour. The second and third outputs supply either 105 or 150 volts, 0–30 ma, regulated. The fourth supplies 6.3 volts a-c, 0–6 amperes, center tapped. The unit has 0–350-volt and 0–300-ma meters.

LITERATURE AVAILABLE

Voltage Regulators. Bulletin S351 gives data on line of automatic voltage regulators. *Superior Electric Co., Bristol, Conn.*

Coatings. Catalog describes liquid anticorrosion and antiabrasion coatings. *Magic Chemical Co., 121 Crescent St., Brockton 2, Mass.*

Signal Generators. Illustrated folder gives data on sweep-signal generators. *Kay Electric Co., 14 Maple Ave., Pine Brook, N. J.*

Potentiometer. Small precision potentiometer is described in catalog sheet. *DeJur-Amsco Corp., Industrial Division, Dept. N-4, Northern Blvd., Long Island City 1, N. Y.*

Stainless Steel Pipe. Data sheets describe stainless steel pipe and fittings. *Carpenter Steel Co., Alloy Tube Division, Union, N. J.*

Test Chambers. Bulletins describe vacuum, temperature, and humidity test chambers. *Tenney Engineering, Inc., 26 Avenue B, Newark 5, N. J.*

Enriched Isotopes. Document Y-772 lists electromagnetically enriched isotopes available as of May 31, 1951. *Oak Ridge National Laboratory, Plant Records Dept., Y-12 Plant, Oak Ridge, Tenn.*

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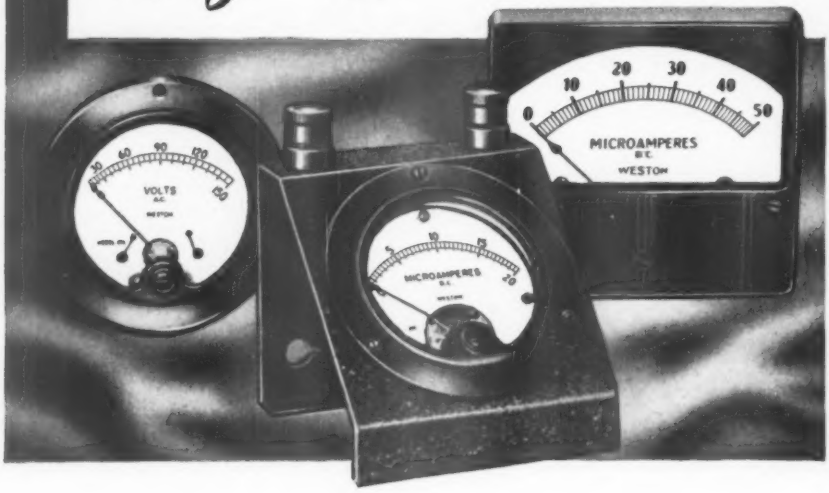

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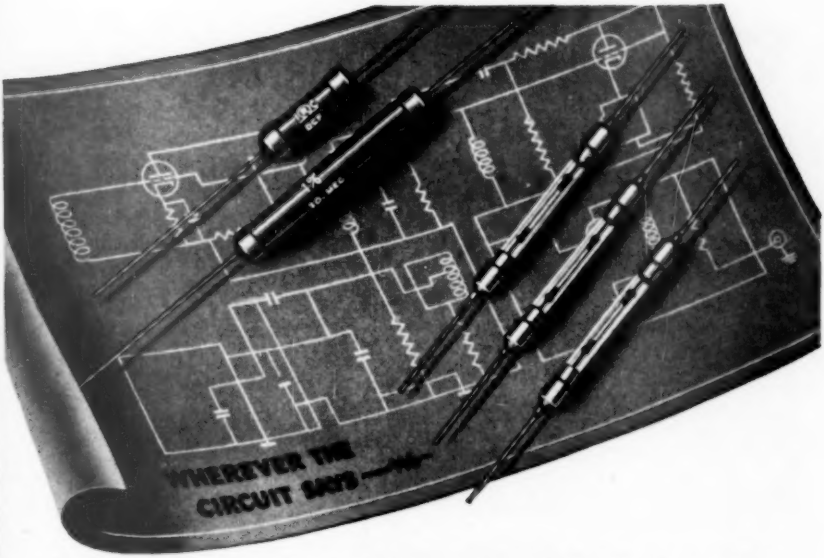

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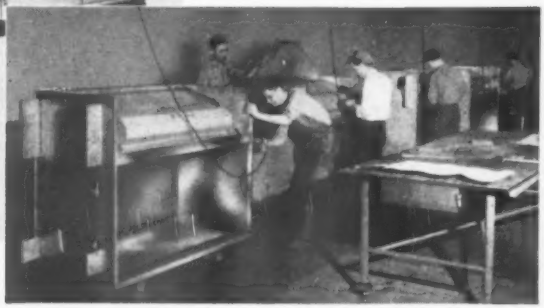

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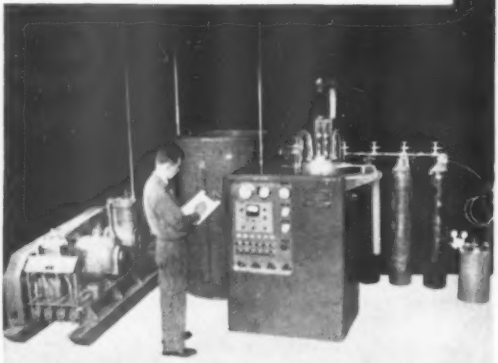

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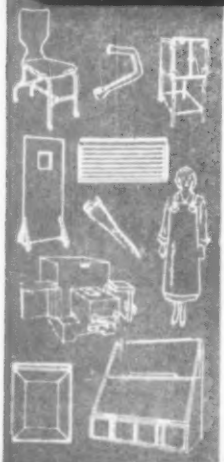

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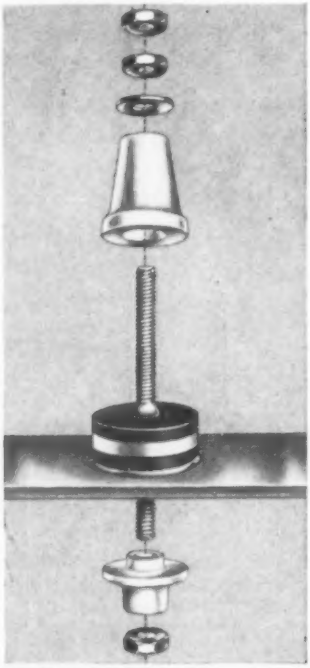

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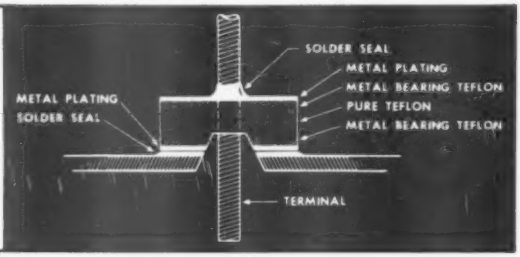

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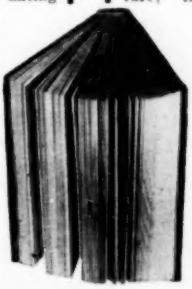

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THE KELLEY-KOETT INSTRUMENT COMPANY

936–8 York Street, Cincinnati 14, Ohio

Leading Manufacturer of Instruments for the Atomic Age

Specifications

ALPHA CHAMBER: Field strength up to 2000 volts/cm for low resolution losses.

AUTOCOUNT: Predetermined register counts of 10, 100 or 1,000 selected by a front panel control.

AUTOTIME: Predetermined timing up to 60 minutes, accurate to 0.1 second (using Keleket Model K-214 timer).

AUTOMATIC RESET: Front panel switch to reset scaling stages and to actuate the automatic motor-driven reset mechanism.

INPUT SENSITIVITY: For the Alpha Chamber, 0.3 millivolt; for the scaler, 0.5 volt.

ATTENUATOR: Logarithmic; 20% increase in sensitivity per step; 24 steps; front panel control.

AUXILIARY OUTLETS: For oscilloscope, automatic timer, standard timer, 110 V. AC.

SIZE: 10½" x 21½" x 15". Chrome plated chamber is 3½" dia. x 6¾" long.

WEIGHT: 60 pounds.

How to find an "Alpha" in a Crowd

NUCLEAR Model 117 Methane Proportional Flow Counter is the ideal instrument for counting alphas in the presence of high beta and gamma activity.

Several complete instruments and accessories are embodied in the compact Model 117: A methane flow proportional counter with variable geometric sample holder; a variable gain, high-gain linear amplifier, with calibrated control; a Higinbotham-type scaling circuit of 256:1; a high-speed impulse recorder; and a variable, stabilized high-voltage supply.

Model 117 was the first commercial instrument of its kind, and has been proven thoroughly reliable in use by leading nuclear scientists.

Complete data on Model 117 will be sent promptly, without obligation, upon request.

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